FOOD TECHNOLOGY

PUBLISHED BY THE INSTITUTE OF FOOD TECHNOLOGISTS

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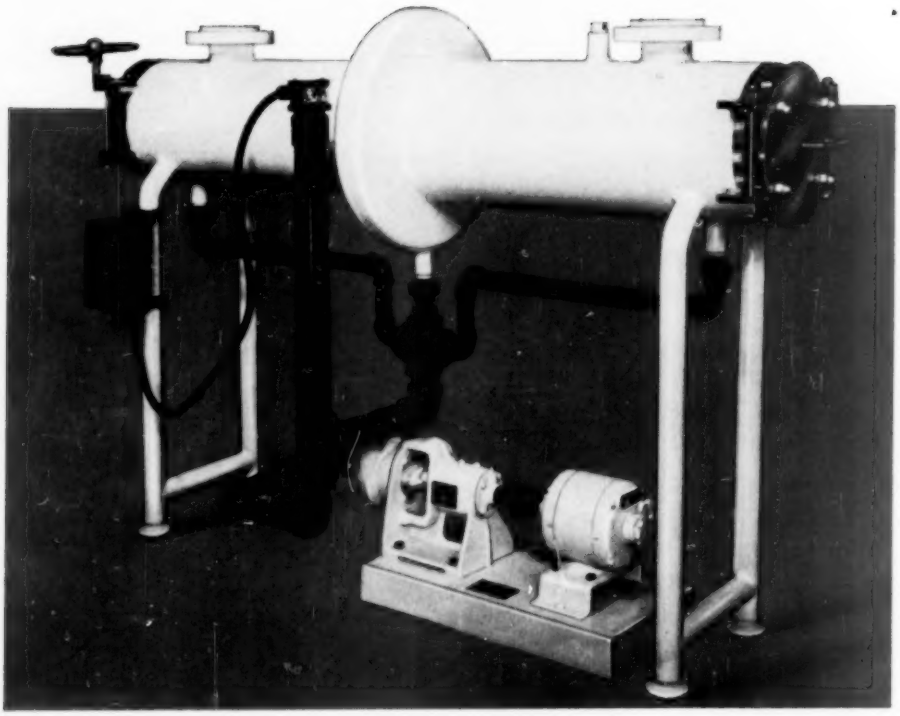

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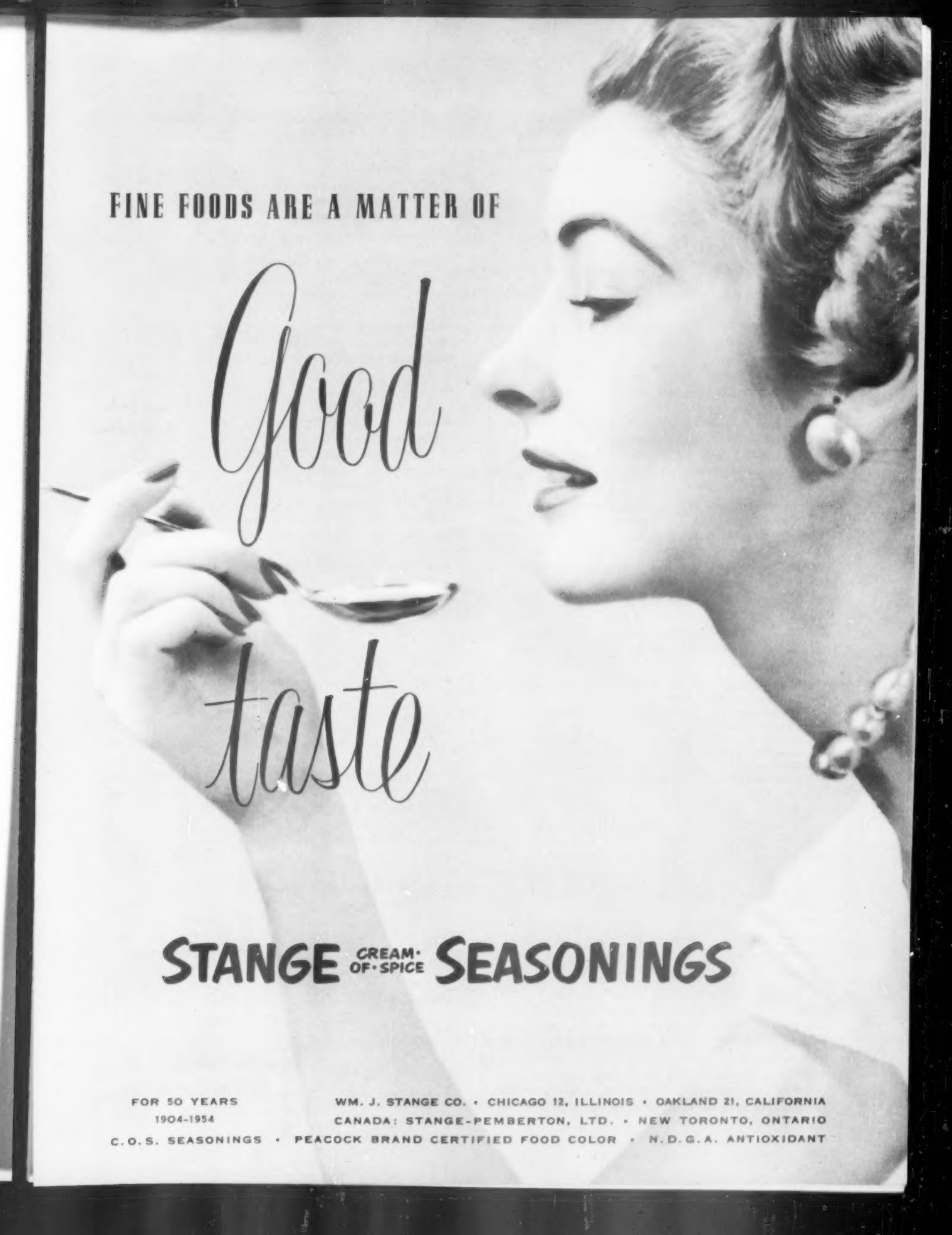
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Affairs of the Institute

News of the Regional Sections
Books
Annual Meeting Bulletin Board
Selected Abstracts
Recently Elected Members
Future Dates for Food Technologists
Food Industry Topics

Regional Section News

MOHAWK VALLEY SECTION

The dinner meetings of the Mohawk Valley Section, Canajoharie, New York, were held as follows:

At the November meeting, Mr. Michael Kubovciak, Bacteriologist of Beech-Nut Packing Company, Canajoharie, New York, presented an interesting topic, "The Growth and Development of the Banana Puree Industry." His discussion was illustrated with movies.

The December meeting gave us the opportunity to hear Dr. Z. I. Kertesz of the New York Agricultural and Experiment Station, Geneva, New York. Dr. Kertesz spoke of his experiences in Ceylon where he was connected with the Food and Agriculture Organization of the United Nations. His stimulating discussion was supplemented by colored slides.

The Section met in Canajoharie, N. Y., on January 14 at the Fort Rensselaer Club. Mr. Allen H. Saul of the Beech-Nut Packing Company, Canajoharie, N. Y., was the guest speaker. His interesting discussion on coffee was illustrated with movies from the Pan American Coffee Films—"Challenge to Coffee in 1954," and "Good Things Happen Over Coffee."

A short business meeting preceded Mr. Saul's discussion. A new slate of officers for the 1954 term was elected as follows:

Chairman: Michael Glickstein, General Ice Cream Corporation, Schenectady 1, New York.
Vice Chairman: Dr. F. M. Skelton, General Ice Cream Corporation, Schenectady 1, New York.
Recording Secretary: Elsie E. Rhodes, Beech-Nut Packing Company, Canajoharie, New York.
Corresponding Secretary: Carl W. Nordgren, Chr. Hansen's Laboratory, Inc., Little Falls, New York.

INDIANA SECTION

The Indiana Section held its winter meeting on January 12th at the Athenaeum Turners Club in Indianapolis. The principal speaker of the evening was Dr. Berton S. Clark, President of the Institute of Food Technologists. The subject of Dr. Clark's talk was, "Current Trends in Metal Containers." There was a very good attendance by the members and many newcomers were introduced to the group by Chairman D. T. Sherow.

PHILADELPHIA SECTION

Dr. R. R. Baldwin of the Central Research Laboratories, General Foods Corporation, Hoboken, N. J., spoke on the use of isotopes in food technology at the February meeting of the Philadelphia Section, held at the Faculty Club, Drexel Institute Dr. Baldwin, in addition to describing the production, availability, cost and uses of isotopes in food technology, briefly reviewed the status of cold sterilization by means of irradiation.

NORTHERN CALIFORNIA SECTION

On February 11 the Northern California Section held a most unusual meeting. C. A. Rietz, President of Rietz Manufacturing Company, spoke on "Foods in Asia," illustrating his talk with color slides. The speech followed a genuine Chinese banquet, held at Kan's Restaurant, in San Francisco.

CHICAGO SECTION

A new type of program was launched with the first meeting of the new year of the Chicago Section. Four food technologists spoke on current research projects. Dr. Joseph E. Bradley, QM Food and Container Institute, Chicago, spoke on "Consumer Bias in Food Preference Testing." He told of the correct words to use when explaining and training taste panels. Using examples, he explained the correct procedures to use when interviewing.

Slides and diagrams were used by Ben Gross, research chemist of The Quaker Oats Co. in his paper on "Dry Corn Milling Research."

"The Effect of Heat Treatment on Enzyme Inactivation and Cloud Stability of Frozen Concentrate Orange Juice" was presented by Dr. Richard B. Guyer, Project Engineer, Continental Can Co. With slides and graphs, he pointed out the work that is being done on Florida pineapple and valencia and pineapple oranges. By regulating amount of heat and time, the desired stability was secured.

Paul W. Hardy, Research Dept., American Can Co. suggested a new field of investigation in his demonstration on "Possible Similarities Between the Human Olfactory and Color Vision Mechanisms." Before launching a new food product, technologists should realize that its color and scent can effect its success when placed at the dining table.

WESTERN NEW YORK SECTION

The record dinner meeting of the Buffalo Chapter, Western New York Section, was held jointly with the Cereal Chemists of Western New York on December 7, 1953. The turn out for this meeting was very gratifying and indicated that a good deal of advance publicity had resulted in this response.

Our speaker was Mr. R. L. Lloyd, Laboratories and Technical Service Dept., American Maize Products, Inc. His topic was: "The Many and Varied Functions of Sugars and Starches in the Food Industry." Mr. Lloyd's talk covered quite adequately the uses of starches in the adhesive industry and in the food industry as well as its use as an additive for bauxite in the initial processing stages to separate out many non-essential elements during bauxite mining operations.

Many pamphlets, leaflets, etc., were distributed at the end of the program relative to the research work that American Maize is carrying on in the food field.

NEW YORK SECTION

What was probably the largest gathering of the New York Section of Institute of Food Technologists came off at its January 20, 1954 Meeting, held in the Brass Rail Restaurant, 43rd Street and Fifth Avenue. With

(Continued on page 7)

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How They Made Margarine a Better Food

by Science Writer

Over a hundred million pounds of margarine are used a month as a table spread, a tasty seasoning for hot vegetables and for many other purposes in cooking and baking. Included in the "Basic 7" food list, it serves as one of the finest and most concentrated energy foods. It is a shining example of a food which has been made repeatedly better.

In the original product which was invented more than 80 years ago, beef fats were used. Later these were replaced by lard and then by vegetable oils. When raw the latter, however, had several drawbacks: an unpleasant odor and taste, unsatisfactory color and a low melting point. These have been overcome by scientific refining methods, hydrogenation and use of certified food colors. Strictly phrased Federal Standards sponsored by the industry assure maintenance of very high quality, uniformity and nutritional value.

With this history of product improvement, it was natural that margarine makers would make their food a more valuable one by the addition of vitamin A. For over a decade many brands of table margarine were marketed with a label statement that they contained 9,000 International Units of vitamin A per pound. Now virtually all brands are fortified with vitamin A and the minimum amount supplied per pound is 15,000 I.U., an increase of over 66%.

Here are some of the benefits which vitamin A supplies. Scientific evidence is available showing that it helps to maintain normal vision and to overcome night blindness; that it increases the body's resistance to infections; that it promotes physical and mental development; and that it aids in maintaining normal glandular function.

Margarine makers who were accustomed to "fishy" odors and flavors when buying vitamin A sought a new source of supply. They found this new source in synthetic vitamin A—as produced by the famous synthesis evolved by the Roche research team headed by Dr. Otto Isler.

It was natural that margarine makers would adopt synthetic vitamin A for fortification of their products. When they were dependent on vitamin A extracted from fish liver sources, they found wide variations in vitamin A price. Synthetic vitamin A eliminated this disadvantage because its price is reasonable and stable. And a second disadvantage disappeared because synthetic vitamin A has absolutely no "fishy" odor, or taste, or after-taste!

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This article, reprints of which are available without charge, is published as a service to the food industry by Hoffmann-La Roche Inc., Nutley 10, New Jersey. In Canada: Hoffmann-La Roche Ltd., 286 St. Paul Street, West, Montreal, Quebec.

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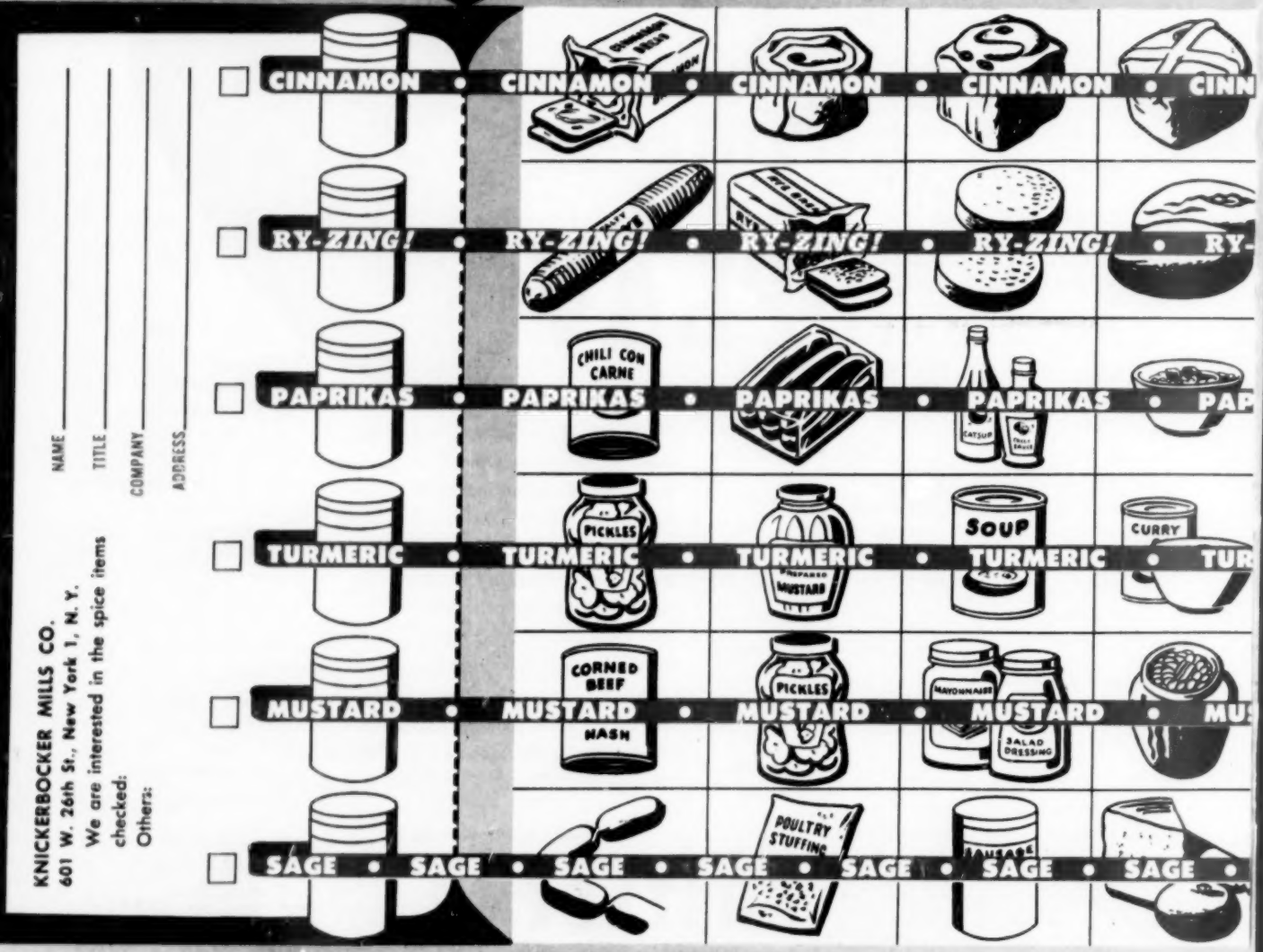
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REGIONAL SECTION NEWS

(Continued from page 2)

approximately 211 present, more than could be squeezed into the dining room for the dinner, it appears that the New York Section may soon be on the hunt for larger accommodations. Thirteen out-of-town visitors were present, including three from California sections.

Dr. L. V. Burton receives from Mr. Al Schaal the commemorative gavel awarded to IFT past presidents.

Presumably the extra-large turnout was due to the announced subject "Egg Solids in '54." The principal part of the program was devoted to a series of technicolor slides devoted to glorification of life in the U.S.A., with scant technical allusion to the egg solids industry. (The music lovers in the audience enjoyed the muted tones of Dvorak's New World Symphony, which accompanied the slides.) Fortunately, Dr. H. M. Slossberg, Technical Director of Research and Production, Henningsen, Inc., was available for the faithful few that remained to ask questions.

Preceding the evening discussions, the New York Section presented an engraved gavel to Dr. L. V. Burton, Executive Director of Packaging Institute to commemorate his services as the second president of IFT.

MARYLAND SECTION

The following officers were elected to serve the Maryland Section for the current year.

Chairman—Arthur M. Kaplan, Microbiology Research Division, U. S. D. A., Beltsville.
Vice-Chairman—LeRoy V. Strasburger, Strasburger and Siegel, Baltimore.
Treasurer—William A. Feild.
Secretary—William J. Hart, Jr., Virginia Polytechnic Institute, Blacksburg, Va.

The January meeting was held at the Stafford Hotel, Baltimore, with 37 members and guests present.

The Speaker of the evening was Mr. A. I. Totten, Jr., Director of Research of the Light Metals Division of the Reynolds Metals Company, Richmond, Virginia. Mr. Totten spoke on "Aluminum Foil—its Present and Future for Food Packaging." He traced the development of aluminum from its recovery by Charles Hall to the present manifold uses in all of its forms. He showed how, in two years, aluminum foil was promoted from decorative to utilitarian use by the housewife. He described the many composite packaging materials that are in vogue utilizing thin foil, wax paper, plastics and resins. It is apparent that we are just at the beginning of newer things in food packages.

GREAT LAKES SECTION

The Section held its Winter Meeting on January 15, 1954, at the Park-Shelton Hotel in Detroit, Mich. An all-day session, the morning was devoted to touring one of the newest and most modern warehouses in the world —that of Abner A. Wolf, Inc., 8601 Meadowdale Ave., Detroit. This plant and the adjacent cold storage warehouse of the Wrigley Stores provided excellent examples of the modern technics in food handling and distribution. The afternoon program was devoted to the following papers which dealt with the vitamin fortification of foods:

"Philosophy of Food Fortification"—Dr. Wilma Brewer, Michigan State College.
"New Developments in Vitamin A Fortification of Food Products"—G. F. Siemers, Hoffman-LaRoche, Inc.
"Reinforcement of Foods with B Vitamins"—C. L. Brooke, Merck & Company.
"Current Investigations in the Fortification of Food Products with Ascorbic Acid"—E. E. Macdonough, Chas. Pfizer, Inc.

Mr. F. H. McIntire, Special Agent in Charge, Detroit Office F. B. I., addressed the evening dinner meeting speaking on "The Work and Functions of the Federal Bureau of Investigation."

ST. LOUIS SECTION

The St. Louis Section was fortunate to have as its guest speaker for the January meeting, the National Secretary, Colonel C. S. Lawrence. The subject of the talk, "Playing Host to a National Meeting" was a most timely one, and of real interest to every member of our organization. If you haven't heard, the St. Louis Section is playing host to the National Convention in 1956.

Colonel Lawrence was also presented with an honorary membershhip card for the local section. He may not have realized it, but this also makes him eligible for membership on the various committees being formed to work on the 1956 National Convention.

The March meeting will be held at the University of Missouri, in Columbia, instead of at St. Louis, and will consist of a two-day meeting on March 19 and 20. The program has been set-up as follows:

FRIDAY, MARCH 19

Morning:
Tour of Campus Facilities Relating to Food Technology.

Afternoon:
Symposium—Technical Problems Involved in the Production of Special Dietary Foods.

Evening:
Banquet and After-dinner Speaker.

SATURDAY, MARCH 20

Morning:
Symposium—Flavor and Odor Evaluation Methods.

Afternoon:
Symposium—Microbiological Problems Involved in Improving Product Acceptability.

(Continued on page 8)

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REGIONAL SECTION NEWS

(Continued from page 7)

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Books

SUBMICROSCOPIC MORPHOLOGY OF PROTOPLASM. A. Frey-Wyssling. Elsevier Publishing Company, Amsterdam, 1953. 411 pp.

This volume is the third edition of the author's "Submikroskopische Morphologie des Protoplasmas und seiner Derivate." Despite its more restricted title, this edition also deals with the submicroscopic structure of protoplasm and its organelles, as well as its derivative products.

As in the second edition, the treatment of the subject is organized into three main divisions: (1) *fundamental aspects of submicroscopic structure,* including organization of sols, crystal structure, structural chemistry, phase, gel structure, and a critique of the methods of investigation and their limitations (polarization microscopy, x-ray analysis, electron microscopy); (2) *fine-structure of protoplasm,* dealing specifically with the cytoplasm, nucleus, chloroplasts, erythrocytes, and gametes; (3) *fine-structure of protoplasmic derivatives,* with particular stress on cellulose, cutin, starch, actomyosin, chitin, silk, keratin, reserve protein, collagen, and nerve structure.

Much of the text remains unchanged from the second edition. However, a general editing has improved the translation. A few illustrations and paragraphs have been omitted from the new volume, and there has been some revision of organization.

The principal contribution of the new edition lies in the author's more comprehensive discussion and evaluation of the techniques and evidence of electron microscopy. New topics have been selected for consideration (gamete structure, bone and tooth structure), and older ones have received more extended treatment (particularly protoplasmic viscosity, cytoplasmic proteins and cyotplasmic structure, chloroplast organization, cellulose cell walls, starch grain structure, and muscle fibers). These are supplemented by graphic electron micrographs, which contribute to the readability of the text. Perhaps one might desire a more extended review of the recent work on mitochondria. This topic seems somewhat slighted in view of the author's program of integrating structure with physiology.

Because of his own major contributions in botany and biophysics, Frey-Wyssling is well able to evaluate the advances in our knowledge of fine-structure and to relate them to the functioning of the organisms. In general, the text is directed, and can especially be recommended, to those who wish to learn something of the significance of this new field in interpreting problems of morphology and physiology. To this end, methods of research and principal results are adequately treated.

(Continued on page 10)

Notes from the makers of
Myvax Vitamin A

Vitamin A
in a shrimp's eye

We used to make vitamin A out of fish liver oils. Europe *still* gets much of its vitamin A from Antarctic whales with the help of radar, explosive harpoons, and highly efficient factory ships.

But, modern as it is, this is still the hard way.

In 1948 we brought out the first man-made vitamin A available in America in commercial quantities. It started something. The whole American industry turned to synthetic vitamin A.

Yet, our scientists keep eyeing those whales. For the whale—and other creatures of the sea—still harbors secrets about vitamin A we'd like to know more about.

We are interested, for example, in the fact that the whales feed interminably on shrimp. Running it back a step, we find that shrimp manufacture vitamin A in their eyes, and in great quantity—as much as 12,000 International Units of vitamin A per gram, dry weight, for large specimens of *Meganyctiphanes norvegica*.

Running it back still another step, the potency of vitamin A oil in these deep sea crustaceans measures two or three times higher chemically than it does biologically. The answer to such an anomaly might some day help all of us obtain better, more versatile synthetics.

So we keep inquiring about it—even though we've left the shrimp and the whale far behind as economical producers of vitamin A.

Vitamin A is a fast-breaking subject. If you use it in your product, it's nice to be able to feel that you won't be caught in the backwash of developments. You enjoy that feeling by keeping in touch with the makers of Myvax Vitamin A and Myvapack Vitamin A—both immediately available from stock so that you can keep your vitamin A inventory at any level you like. ***Distillation Products Industries,*** Rochester 3, N. Y. (Division of Eastman Kodak Company). Sales offices: New York and Chicago • Charles Albert Smith Limited, Montreal and Toronto • W. M. Gillies and Company, Los Angeles and San Francisco.

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Program for the Ladies, IFT Meeting in Los Angeles. Marjorie Heid, Chairman, has just turned in an advance copy of the program for IFT ladies. It follows:

SUNDAY, JUNE 27

Welcome Tea and get-acquainted social in honor of Mrs. Clark and Mrs. Bates. To be held in the Biltmore Hotel.

MONDAY, JUNE 28

Farmer's Market — Ladies will visit the many different shops for which the Market is so famous; then each one will purchase her own lunch from one or more of the food stalls, and they will all eat together on the sun deck.

Motion Picture Studios— Tanner Bus Tour. The ladies will be taken in the same busses to the Market then on to the following studios—Columbia Ranch, Universal, Walt Disney and Republic.

TUESDAY, JUNE 29

Trip to Laguna Beach—The busses will take the ladies through the beautiful citrus groves of Orange County and along the scenic coastline of Balboa and the lovely beach homes on Balboa Bay and Corona del Mar.

Laguna is built along the ocean upon high cliffs and on rolling hills, which makes it one of the most charming of our beach resorts.

Luncheon will be served at picturesque Victor Hugo Restaurant overlooking the ocean. The Artists will have a special sidewalk exhibit, and the many gift and pottery shops, for which Laguna is known, will welcome the ladies.

If they desire, they may partake of clam chowder made on the beach for them exclusively.

WEDNESDAY, JUNE 30

Trip to Knott's Berry Farm with the men.

THURSDAY, JULY 1

This day is left open for the ladies to shop in our many delightful stores.

New Registration Technique Will Save Annual Meeting Participants from Standing in Line. At Los Angeles, June 27-July 1, 1954, representatives of all the technologies known to the food field will gather to compare notes. It is already evident that this opportunity to integrate ideas in food research will draw a heavy attendance. National IFT members are therefore being given an opportunity to register in advance for the Los Angeles meeting and to reserve tickets for all scheduled functions. This plan will be easier on the feet, back muscles, and patience of the registrants. The pre-registration procedure will also give members priority for tickets to events of limited attendance. It is desired that this innovation in Annual Meeting procedures be brought to the attention of as many food technologists as possible. The plan works as follows:

During the next few weeks each National member will receive through the mail an application for registration and tickets to the Social Hour, the outing at Knott's Berry Farm, banquets, plant trips, and other events on a program that promises to be an invaluable professional experience as well as a travel experience second to none. The Registration Committee, upon return of the application and payment for registration and chosen social functions, will acknowledge receipt of the application by an acknowledgment postal card which will serve as identification for the distribution of badges and tickets in Los Angeles at the time of the Annual Meeting. The postal card will provide space for the member to fill in his Los Angeles address so that the meeting roster may include the members' permanent as well as his temporary address. This will help members to get in touch with their friends immediately on arrival. Upon presentation of the acknowledgment postal card at a special PRE-REGISTRATION DESK, each member will be able to pick up his badge, enscribed with his name and affiliation as well as a complete book of tickets for his selection of events without delay. Each ticket obtained through *pre-registration* will have the member's name imprinted, thereby facilitating recovery in case of accidental loss.

No Registration Fee is being charged for wives of registrants. However, members' families will be requested to register in order to qualify for tickets to the Social Hour, the outing at Knott's Berry Farm, and the other events scheduled especially for the ladies. One such event is a trip to beautiful Laguna Beach, its art colony, and the renowned Victor Hugo Restaurant with its flower gardens running down to the sea. The deadline for Pre-registration will be June 15, 1954. All members are urged to return their application for registration and tickets as far in advance as possible since tickets to some of the functions will be necessarily limited. Registration Fees have been set at $10.00 for Members and Exhibitors, $15.00 for Non-members, and $3.00 for Students. Pre-registration will permit more orderly, efficient, and convenient registration. In fact, it does away with one of the little irritations of all conventions—waiting in the waiting line while you could be talking with friends you haven't seen for a year.

BOOKS

(Continued from page 8)

Numerous illustrations effectively supplement textual descriptions and concepts. The extensive bibliography is especially useful in that the subject of the publication (if not the title) is indicated for each reference.

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Selected Abstracts*

ANALYTICAL METHODS

Determination of moisture in peanut butter.

PEPPER, M. B., AND FREEMAN, A. F. *J. Am. Oil Chemists' Soc., 30,* 335-7 (1953).

The moisture and volatile content of whole peanuts was detd. by A. O. C. S. Official Method Ab 2-49 and was compared with the moisture content of the sliced peanuts detd. by a toluene distn. procedure described by Tryon. For practical purposes the results obtained by these methods are in agreement.

Determination of moisture in industrial and commercial practice.

KLOCKMANN, R. *Fette u. Seifen, 55,* 52-3 (1953); *Chem. Abstr., 47,* 10407e (1953).

A com. app. for detg. H_2O with an accuracy of $\pm 3\%$ is described. It consists of a metal pressure bottle with attached pressure gage into which the finely divided sample and an ampule of CaC_2 are placed. The bottle is then shaken to break the ampule and the increase in pressure due to formation of C_2H_2 is measured and the H_2O content calcd.

The odors of optical isomers.

POSVIC, H. *Science, 118,* 3589-99 (1953).

Certain pairs of optical antipodes displaying different odors were cited as arguments against the Beck-Miles infrared theory of olfaction. The d- and l-dimethyl *trans* hexahydrophthalates were chosen for investigation because they seemed to offer the greatest possible freedom from chem. difficulties. These samples had odors which were indistinguishable qualitatively and seemed to be of comparable intensity. d,l-Limonene was also examd. and it was found that it differed in odor from d-limonene due to an impurity present. It is concluded that 2 optical isomers have not been shown to have different odors.

An effective safety pipette.

RODKEY, F. L. *Science, 118,* 488-9 (1953).

A simple, inexpensive app. which attaches to a pipette to prevent toxic or hazardous liquids from contacting the operator is described. The liquid is drawn into the pipette by rubber bulb contg. a glass bead stopper. It is claimed that one can accurately deliver any desired vol. of liquid.

Automatic volume fraction collector.

Anal. Chem., 25, 1423 (1953).

Six different automatic fraction collectors for chromatographic or distn. work are described.

A micro lipid extractor.

MITCHELL, P. *Nature, 172,* 124 (1953).

An app. designed to make possible the quant. extn. of phospholipid on a microscale is described. Best reproducibility was obtained on 5-50 mg. of material contg. 0.3 to 5 mg. of lipid.

BIOLOGICAL SCIENCES

BIOCHEMISTRY

How is a protein made?

LINDERSTROM-LANG, K. U. *Sci. American, 189,* 100-6, September, 1953.

A discussion of protein synthesis which includes special consideration of the sources of energy involved.

Radiation chemistry of cysteine solutions.

WHITCHER, S. L., ROTHERAM, M., AND TODD, N. *Nucleonics, 11,* 30-3, August, 1953.

A study of some of the effects of irradiation on the amino acid cysteine.

Physiology of the D-amino acids.

BERG, C. P. *Physiol. Revs., 33,* 145-89 (1953).

A review of the intermediate metabolism of the D-amino acids including growth response and excretion studies. 380 references.

Radioactive measurement of proteolytic activity.

NARDI, G. L. *Science, 118,* 299-300 (1953).

A procedure for direct quant. estn. of proteolytic activity was devised. The principle of the method depends upon the digestion of I^{131}-labeled albumin by a proteolytic enzyme. This will result in formation of unbound radio iodine that, though not necessarily in inorg. form, is no longer attached to the whole protein and may be sepd. from the latter by selective pptn. of the albumin. The radioactivity contained in the supernatant should then be proportional to the proteolytic activity of the enzyme.

Effects of acidosis on the metabolism of fructose.

MACKLER, B., AND GUEST, G. M. *Am. J. Physiol., 174,* 54-6 (1953).

Intravenous fructose tolerance tests were done on dogs in normal (fasting) states and in states of severe acidosis induced by intravenous perfusions of NH_4Cl soln. Results of these tests indicated that fructose disappeared at the same rate from the blood of non-acidotic animals and acidotic animals. Results of studies on the metabolism of fructose in blood *in vitro* indicate that fructokinase is probably absent from blood, and that acidosis inhibits the utilization of fructose by blood cells.

D-enzyme: a disproportionating enzyme in potato juice.

PEAT, S., WHELAN, W. J., AND REES, W. R. *Nature, 172,* 158 (1953).

An enzyme in the potato which can effect the synthesis and degradation of (short) amylose-type chains by transglucosylic action, was found assocd. with the Q-enzyme. This new "D"-enzyme brought about rapid disproportionation of malto-triose, -tetrose, -pentose, -hexose, and -heptose. The products were unbranched α- 1:4 chains. Maltose was neither utilized nor formed by the action of D-enzyme.

A simple method for the preparation of crystalline potato phosphorylase and Q-enzyme.

BAUM, H., AND GILBERT, G. A. *Nature, 171,* 983-4 (1953).

The procedure is described in detail and consists essentially in adsorbing the enzymes from alc. soln. on pea amylose.

Kinetic analysis of enzyme reactions. I. Further considerations of enzyme inhibition and analysis of enzyme activation.

SEGAL, H. L., KACHMAR, J. F., AND BOYER, P. D. *Enzymologia, 15,* 187-98 (1952); *Chem. Abstr., 47,* 5464g (1953).

The equations for enzyme inhibition obtained by the Michaelis-Menten treatment are reexamd. by steady state kinetics and further extended to include nontotal inhibition. Equations for the rates of enzyme reactions involving an activator, coenzyme, or second substrate are derived for both essential and nonessential activation. It is shown how kinetic data can be utilized to test the validity of postulated reaction mechanisms.

NUTRITION

The psychology of eating.

Proc. Nutrition Soc., 12, 143-65 (1953).

A symposium containing the following papers: Psychiatric implications of disturbances of eating and nutrition, Millar, W. M. (144-8); Peptic ulceration, Illingworth, C. F. W. (148-53); Anorexia nervosa, Rolland, C. F. (153-6); Physiological effects of smoking, Walker, J. M. (157-60); The effect of lesions in the hypothalamus on appetite, Kennedy, G. C. (160-5).

Infant feeding.

BAKWIN, H. *J. Clin. Nutrition, 1,* 349-54 (1953).

Infant feeding is reviewed in the light of current knowledge about child development. Breast-feeding is recommended. Aside from the psychological implications, which may be significant in individual instances, breast- and bottle-fed infants show definite somatic differences in chem. constitution, in metabolic response (to lactose and to ascorbic acid administration), and in resistance to disease. A modified self-demand feeding is recommended which relates to the amt. and, later on, the types of food, the intervals between feedings being kept fairly const. In giving advice for infant feeding, it is fully as important to consider the personality of the mother as the child. The optimal time to intro-

(Continued on page 14)

* These Selected Abstracts are made available to FOOD TECHNOLOGY through the cooperation of Associate Editor H. A. Campbell and the General Foods Corporation of New York, N. Y. The abbreviations found in these abstracts are similar to those used by Chemical Abstracts.

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SELECTED ABSTRACTS

(Continued from page 12)

duce solid foods is when the infant is developmentally ready, as shown by a change in oral behavior, about 12 to 16 weeks.

The regulation of food intake after hypothalamic lesions.

Nutrition Revs., 11, 55-7 (1953).

The glucostatic theory of the regulation of food intake provides an explanation for the hyperphagia following hypothalamic lesions. To gain widespread acceptance of this view addnl. exptl. evidence must be obtained elucidating the mechanism whereby the blood sugar of these operated animals remains low despite increased food intake.

Nutritive requirements of the cat: a preliminary report.

Morris, M. L. *Vet. Med., 48,* 451-6 (1953).

The trends which further investigations should take are indicated on the basis of a review of work done thus far. An attempt is made to tabulate the large quantity of contradictory information available.

Corn meal enrichment.

Lease, E. J. *J. Am. Dietet. Assoc., 29,* 866-71 (1953).

The enrichment of corn meal and grits in the southern part of the U. S. is becoming more widespread. In South Carolina, enrichment of all types of corn meal and grits that are sold has been mandatory for several years. Clemson Agricultural College compounds an enrichment mixt. for whole corn meal and distributes it to small corn mills in the South. This college also has designed and is manufg. and distributing enrichment equipment suitable for such mills. These services are rendered on a non-profit basis and are coordinated with an eduactional program to encourage millers to enrich their corn meal and grits. Enrichment of corn meal offers an inexpensive method of eliminating pellagra and improving the diets of people in those parts of the world where corn is a staple food.

Antibiotics in nutrition.

Braude, R., Kon, S. K., and Porter, J. W. G. *Nutrition Abstr. & Revs., 23,* 473-95 (1953).

A review with 372 references. Although no spectacular bacteriol. change occurs when these small amts. of antibiotics are given to animals, there is no doubt that the gut population will adapt itself in the presence of the antibiotic with danger of the development of noxious resistant strains or of the gradual loss of activity of the antibiotic through the development by the gut flora of resistance to its action. In Great Britain the addn. of antibiotics to rations is for the time being prohibited. This is being amended so that antibiotics will be available for use in animal feeds, but as the cost of such supplements is not yet known it is impossible to assess the precise economic advantage from their use, though it is reasonable to expect a small margin of profit.

Acetate and pyruvate metabolism in obese mice.

Nutrition Revs., 11, 88-9 (1953).

Obese mice oxidize a smaller proportion of administered acetate than do the nonobese animals and the acetate not oxidized appears to a large extent as body fat. This enhanced rate of lipogenesis may be important in the cause of this type of obesity. On the basis of the incorporation of C^{14} into the expired CO_2, it appears that the obese animals utilize the acetate for purposes other than oxidation to a greater degree than do the nonobese mice. While diminished recovery of the administered C^{14} in the expired CO_2 was also observed in the labeled pyruvate-treated obese mice, the decrease was less significant than that found for the alloxan-treated nonobese mice.

Treatment of obesity with calorically unrestricted diets.

Pennington, A. W. *J. Clin. Nutrition, 1,* 343-8 (1953).

Restriction of carbohydrate alone, appears to make possible the treatment of obesity on a calorically unrestricted diet composed chiefly of protein and fat. The limiting factor on appetite, necessary to any treatment of obesity, appears to be provided by increased mobilization and utilization of fat, in conjunction with the homeostatic forces which normally regulate the appetite. Ketogenesis appears to be a key factor in the increased utilization of fat. Treatment of obesity by this method appears to avoid the decline in the metabolism encountered in treatment by caloric restriction. Details of diet and regimen are given.

Fat-loading studies in relation to age.

Herzstein, J., Wang, C.-I., and Adlersberg, D. *Arch. Internal Med., 92,* 265 (1953); *J. Am. Dietet. Assoc., 29,* 1132 (1953).

Two groups of fasting subjects were given a test meal of 40% cream, to provide 1 g. fat for each kg. of body wt. The mean ages of the groups were 24.9 and 62 years. After 2 hr. the mean rise in total serum lipids for both groups was the same. However, the concn. of total lipids in the serum of the younger group fell almost to the fasting level by the 6th hr., while the older group continued to show a higher level for the 24 hr. period.

PHARMACOLOGY AND TOXICOLOGY

Action of caffeine on gastric juice.

Lahiri, S. C. *Antiseptic (Madras) 50,* 20-1 (1953); *Chem. Abstr., 47,* 6038h (1953).

Caffeine Na benzoate (I), 15 g., injected intramuscularly stimulated the secretion of gastric HCl in 15 min. This was followed by a steep fall in the secretion. I, 15g., administered orally effected a still more prolonged secretion of gastric HCl. It is concluded that frequent and excessive drinking of beverages rich in caffeine, especially on an empty stomach, can produce gastritis and may lead to gastric ulcer.

Metabolism of and tolerance to adipic acid.

Lang, K., and Bartsch, A. R. *Biochem. Z., 323,* 462-8 (1953); *Nutrition Abstr. & Revs., 23,* 615, No. 3297 (1953).

Rats were fed on a standard diet of 80% wheat groats and 20% whole milk powder for periods up to 33 weeks and were given in the food adipic acid, neutralized with NaOH, up to 800 mg. daily. Less than 400 mg. was tolerated without apparent injury. With 400 mg. there was no disturbance of growth or other apparent external effect, but slight histological changes were found in liver and kidney, and more severe changes in the intestinal mucosa. Daily administration of 800 mg. caused diarrhea, changes in the coat and general appearance of the animals, and significant retardation of growth. With a low-protein diet (11%) consisting entirely of wheat groats and 0.2 ml. cod liver oil per rat daily, 400 mg. adipic acid retarded growth. The intolerance to higher amts. of adipic acid apparently depends mainly on injury to the mucous membrane of the intestine, which shows chronic inflammation. Histological changes in other organs are slight. A balance expt. of 14 days showed that excretion of adipic acid was the same whether or not the animals were inured to its intake. The organism did not acquire the power to metabolize increased amts. of the substance.

The toxicity of chlordane vapors.

Ingle, L. *Science, 118,* 213-14 (1953).

Swiss albino mice were placed in a treatment chamber and subjected to 14 days of continuous exposure to a current of air which had first passed through 105 ml. of chlordane in a satn. train. No deaths occurred nor did any mice show signs of anorexia, blindness, or loss of coordination. At autopsy, organs and tissues were normal. Other workers previously found chlordane vapors to be toxic to mice. Further tests indicated that such toxic effects were due to an unreacted intermediate impurity in the chlordane.

Consideration of dose-weight relationships.

Rall, D. P., and North, W. C. *Proc. Soc. Exptl. Biol. Med., 83,* 825-7 (1953).

The toxicity of α-naphthyl thiourea for both wild pigmented and lab. albino rats, when expressed as a dose per animal, ap-

(Continued on page 16)

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SELECTED ABSTRACTS

(Continued from page 14)

pears to be const. over a wide age and wt. range. The use of the customary mg./kg. correction for body wt. introduces marked systemic variation. The requirements for a proper correction factor of dose for wt. of animal are considered and it is suggested that this factor should minimize variation in effective dose between groups of animals of different wts. and make corrected dose independent of body wt.

Tolerance, toxicity, and caloric availability of intravenously injected oxypolygelatin solution in rabbits.

Detrick, L. E., Debley, V., and Haley, T. J. *J. Am. Pharm. Assoc., Sci. Ed., 42,* 633-6 (1953).

Eleven consecutive daily intravenous injections contg. 5% oxypolygelatin (OPG) in a nutrient soln., each equiv. to 7.7% of the body wt., produced phys. deterioration and illness in rabbits and a comparatively low mortality ratio.

PHYSIOLOGY AND MEDICINE

Intravenous infusion of blood proteins of cattle in man.

Gruning, W., and Wissfeld, H.-J. *Beitr. klini. Chir. (Bruns) 186,* 400-18 (1953); *J. Am. Med. Assoc., 153,* 981-2 (1953).

"Adaequan," a proprietary blood substitute protein prepn. obtained from the blood of cattle, was given a therapeutic trial by intravenous infusion to 20 patients who had major operations on the abdomen or thorax. Adaequan, which, according to the information obtained from the manufg. firm, consists predominantly of albumin and only small addnl. amts. of globulin, proved to have considerable oncotic activity and consequently may be recommended for patients who require restoration of the fluid reserve. A mild increase in the *N* elimination in the urine occurring in the first 3 days after the administration of Adaequan was considered as a manifestation of the gradual breaking up of the transmitted albuminous bodies. No conclusions could be drawn concerning the biol. utilization of the animal proteins. None of the patients showed kidney impairment for which the protein foreign to the species could have been responsible. The results suggest that animal plasma, provided that it had been adequately prepd. may be used as a blood substitute for direct intravenous infusion in man.

Clinical use of fat injected intravenously.

Van Itallie, T. B., Waddell, W. R., Geyer, R. P., and Stare, F. J. *Arch. Internal Med., 89,* 353-7 (1952); *Chem. Abstr., 47,* 5561d (1953).

Fat emulsions for intravenous use make possible the convenient administration of a large supply of cals. in a small vol. of fluid. Such emulsions are nontraumatic to veins, and virtually no portion of their content of fat is excreted in the urine or feces. No evidence of damage to the liver or of disturbance in the integrity of the red cell membrane was found in preliminary studies directed toward these problems.

Excretion and retention of two modified gelatins in normal human beings.

Shiteri, P. K., and Highland, G. P. *J. Appl. Physiol., 5,* 814-20 (1953).

Oxypolygelatin and fluid gelatin were given as a 500 ml. intravenous infusion in 12 convalescent patients. At the end of 6 hrs., fluid gelatin showed a significantly smaller urinary excretion and greater plasma retention than OPG. The greatest urinary excretion of both materials occurred within the first 6 hrs. Untoward effects were not noted. A method for accurately measuring gelatin by means of its hydroxyproline content is presented.

Failure of phosphorylated hesperidin to influence fertility in rodents.

Millman, N., and Rosen, F. *Science, 118,* 212-3 (1953).

In contrast to findings by other workers, M. and R. did not observe an anti-fertility action of phosphorylated hesperidin in rats or mice. Even when the lowest oral feeding level was exceeded by a factor of 5 times the daily mg./kg. level given in human trials there was no interference with pregnancy through the oral or intraperitoneal routes.

DETERGENTS AND SOAPS

Detergents for radiological decontamination.

Snell, F. D., Segura, G., Stymen, S., and Snell, C. T. *Soap and Sanit. Chemicals, 29,* 42-4, October, 1953.

Soaps and syndets vary in their efficiency in removing radioactive soil from a particular surface. With both soaps and syndets, the effectiveness is significantly increased by combination with two parts of "Calgon" as sequestrant. Ease of removal varies greatly with the nature of the surface. In the event of an atomic attack, large-scale radiological decontamination would be greatly facilitated by the ready availability of common detergents and of "Calgon" in the homes and at near-by retail stores.

Germicides for soap.

Innes, W. *Soap and Sanit. Chemicals, 29,* 42-5, September, 1953.

A review of recent papers covering the use of D. C. M. X. and hexachlorophene.

ENGINEERING AND PLANT EQUIPMENT

New catalyst removes coffee roasting smoke.

Tea & Coffee Trade J., 105, 50-1, August, 1953.

The catalytic-coating alumina and Pt alloy on the surfaces of the Oxycat rod permits combustion of coffee fumes at a temp. of 500-550° C., much lower than the temp. needed for direct flame incineration. It is estd. that the gas required to remove coffee fumes by incineration would be half as much as that needed to roast the coffee alone. The gas bill for a catalyst installation is only half that for incineration. The result is a completely odorless, invisible smoke with no traces of coffee vapors.

Cathodic protection can save chemical process equipment.

Sudrabin, L. P. *Chem. Eng., 60,* 196-200, May, 1953.

A summary of the know-how of cathodic protection to date. It discusses the IR relationships of natural corrosion cells and the neutralizing of these effects by cathodic protection. Anode selection and various industrial applications are described.

Design of hoppers for use.

Lee, C. A. *Chem. Eng., 60,* 194-5, May, 1953.

A short history of hopper design is presented. Some of the factors to be considered in hopper design including a more theoretical discussion of arching, wedge of max. thrust, etc., are discussed. Hopper design is dependent upon the type of material to be used and the function to be played by the hopper in the system.

Factors influencing the properties of spray-dried materials.

Duffie, J. A., and Marshall, W. R. *Chem. Eng. Progr., 49,* 480-6 (1953).

The bulk d. of spray-dried materials was found to decrease with an increase in drying-air temp. Increasing the feed temp. may cause an increase or a decrease in the bulk d., the effect being small in either case. Increased feed temp. causes a slightly increased drying rate and influences the particle size from atomization due to viscosity lowering. The effect of solids concn. may produce an increase or decrease in bulk d., depending largely on whether the increased feed concn. affects the tendency of the particles to expand.

High-frequency cooking.

Pircon, L. J., Loquercio, P., and Doty, D. M. *J. Agr. Food Chem., 1,* 844-7 (1953).

(Continued on page 18)

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Alva FLAVORS

SELECTED ABSTRACTS

(Continued from page 16)

Equipment is described and cost analysis is shown. Steam is cheaper for small batches with the comparative cost for electricity falling with larger batches. Emphasis is on high-frequency heating as a unit operation in meat processing.

FOOD AND FOOD TECHNOLOGY

DEHYDRATED FOOD

Dehydrated food: chemistry of browning reactions in model systems.

HODGE, J. E. *J. Agr. Food Chem., 1,* 928-43 (1953).

Some relationships are shown to exist among the carbonyl-amino, the nonamino, and the oxidative types of browning. Recent findings provided the basis for an integration of the several isolated partial theories of browning (Maillard, sugar fission, ascorbic acid, furaldehyde) heretofore proposed. The significance of the occurrence of the Amadori rearrangement in the Maillard reaction is stressed, and a mechanism for browning in sugar-amine systems based upon the rearrangement is outlined. Attention is directed to the little-studied but important role of dehydrogenated reductones in both enzymatic and nonenzymatic browning reactions. Extensive bibliography included.

FEEDINGSTUFFS

Chicks need vitamin K when fed antibiotics.

Science News Letter, 64, 249 (1953).

Vitamin K is necessary for normal blood clotting and chicks synthesize it in their intestinal tract. At the same time growth stimulating and other drugs affect the vitamin K normally synthesized. Terramycin and arsonic acid prolonged the blood clotting time significantly, and arsanilic acid might do so.

Latest information on methionine.

FINKLE, D., COUCH, J. R., COMBS, G. F., AND HARRIS, L. E. *Feed Age, 3,* 36-42, October, 1953.

A review article. The latest information on the use of methionine in manufd. feed is presented. Methionine is now available at an economic level that makes it highly practical and desirable for poultry and turkey feeds. Tests show methionine improves swine, dog and fur-bearing animal rations.

Pup growth sparked with amino acids.

Science News Letter, 64, 82 (1953).

Pups will grow to be frisky on a low-protein diet (12%) supplemented with lysine and methionine as well as with a higher protein diet (20%) without the two amino acids. Although lysine alone with the low-protein diet allowed normal growth the addn. of methionine made for more efficient use of the protein.

FISH

Setting phenomenon of fish muscle. I. Influence of electrolytes.

MIGITA, M., AND OKADA, M. *Bull. Japan Soc. Sci. Fisheries, 18,* 117-23 (1952); *Food Sci. Abstr., 25,* 400, No. 1868 (1953).

Fish muscle (from horse mackerel), ground with an adequate amt. of salt, forms a sticky paste, which, on standing, becomes an elastic gel. Salts can be divided into 3 groups with respect to their effect on this reaction: with those of group 1, the mixt. remains a sticky paste; with those of group 2, the reaction proceeds to the formation of an elastic gel; with those of group 3, the proteins are coagulated, with simultaneous hydration. The results are considered to indicate that a gel is formed when the muscle protein is strongly hydrated, and that salts affect the extent of hydration.

GELATIN

Increase of resistance to swelling on conditioning gelatin in atmospheres of high relative humidity.

JOPLING, D. W. *Research (London), 6,* 27S-9S (1953); *Chem. Abstr., 47,* 9108g (1953).

Dry gelatins conditioned at 70% relative humidity develop greater resistance to swelling than those conditioned at lower relative humidities, in support of the view of Bradbury and Martin that the gelatin mols. become more mobile at about 70% relative humidity. Coatings of 3.5% solns. of 3 gelatins on glass plates were dried to about 10 μ thick layers, of measured thickness. After 1 to 8 days at 20° and at a series of relative humidities from 20 to 98, vertical-swell curves were measured in distd. H_2O.

STARCHES

The determination of fat and the effect of extraction on the properties of starches.

LINDEMANN, E. *Starke, 3,* 141-7 (1951); *Food Sci. Abstr., 25,* 332, No. 1590 (1953).

The fat content of com. samples of potato, maize, milo, wheat, and rice starch was detd. The methods used were: (1) extn. with solvent (e.g. petr. ether, ethyl ether, CCl_4 or MeOH) and (2) acid hydrolysis. With the cereal starches, the highest values were obtained after acid treatment, the lowest after extn. with petr. ether, ethyl ether, or CCl_4; with potato starch, similar results were obtained in all the methods used. Unlike cereal starches, potato starch contains practically no fatty-amylose complexes; after treatment with a 10% methanolic soln. of palmitic acid, potato starch takes up fatty acids which can no longer be extd. with hydrocarbon solvents. As a standard procedure, L. recommends a 6-hr. extn. with petr. ether. The quant. compn. of the extd. fats (unsaponifiable matter, sterols, and phosphorus compds.) showed that potato starch had the highest values, followed by wheat starch and the other starches. Compn. varied greatly, however, according to the origin of a given sample. The effect of extn. on milo starch was examd. viscosimetrically and by the pudding test. With decreasing fat content, the results of the pudding test improved.

PHYSICAL RESEARCH

The stability of elementary emulsion drops and emulsions.

COCKBAIN, E. G., AND MCROBERTS, T. S. *J. Colloid Sci., 8,* 440-51 (1953).

The rates of coalescence of oil and water drops at the oil-water interface were measured in the presence of soaps, cholesterol, saponin, serum albumin, and polyvinyl alc. Interfacial viscosity and tension data were obtained for some of the systems. It is concluded that the main factor detg. stability in all the systems examd. is the resistance to wetting of segments of the adsorbed film by the discontinuous phase. An explanation is given for certain empirical rules relating emulsion-type to preferential wettability or soly. of the stabilizing agent in oil and water.

Particle size: its measurement and importance in the chemical industry.

FORSYTH, T. L. *Mfg. Chemist, 24,* 286-91 (1953).

A review of principal methods.

Sedimentometric analysis for the determination of the distribution of particle size of powder.

SUITO, E., AND ARAKAWA, M. *Bull. Inst. Chem. Research, Kyoto Univ., 22,* 7-17 (1950); *Chem. Abstr., 47,* 7263h (1953).

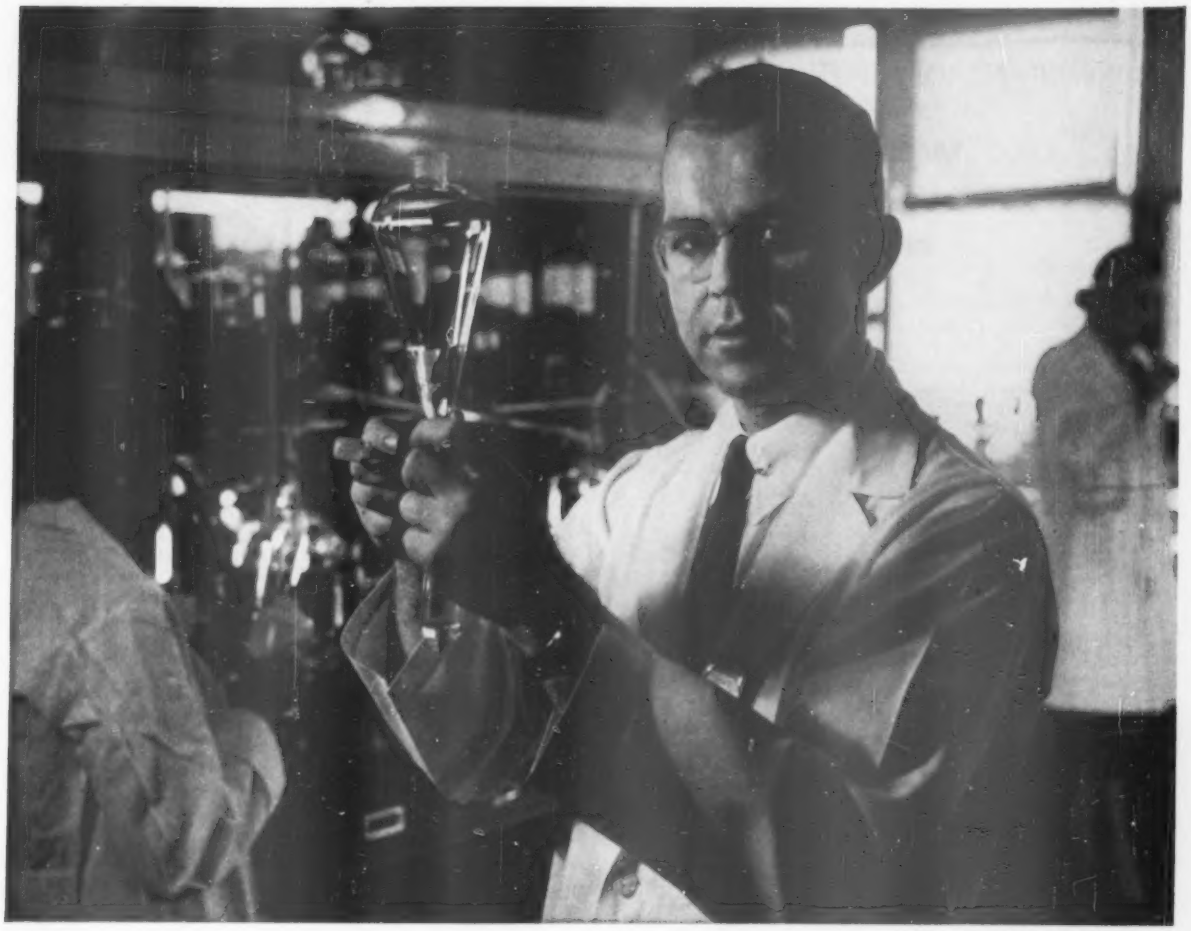
An automatically recording sedimentation balance is introduced, and the theory and practical use with some applications to chem. industry are described. The app. is usable for detg. the distribution curve of particle size of 0.5-40 μ.

The change in the hydrophilic properties of the surface of high-molecular substances during the process of dry grinding.

BARAMBOIM, N. K. *Legkaya Prom., 10,* No. 4, 22 (1950); *Chem. Abstr., 47,* 9109d (1953).

Natural and artificial protein substances were placed in a 2-phase mixt. of water and toluene before and after dry-grinding, and the phase in which they collected was noted. Materials tested include: untanned and chrome-, tannin-, and formalde-

(Continued on page 24 following technical papers)



PHOTOGRAPH BY RICHARD BEATTIE

What we still don't know about cancer

—and one of the reasons why

IN THE PAST FEW YEARS, our knowledge of the nature of cancer, and how to treat it, has grown encouragingly. Patients, who would have been considered hopeless cases even five years ago, today are being completely cured. And even those who apply for treatment too late can usually live longer—and less painfully—because of modern palliative treatment.

All the same, there have been defeats as well as victories. We do not know—to take a single example—why so many more men are now dying from cancer of the lung. In 1933—just twenty years ago—lung cancer killed 2,252 men; in 1953, some 18,500. That's a great increase—which even our expanded population, and other known factors, can't possibly account for in full.

Well, why haven't we found more of the answers to cancer?

Not only because cancer is an immensely complex problem: difficult to diagnose, and difficult to treat; challenging to the best research minds.

All that is true enough. But there is another reason: *we do not* have enough money.

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You gave the Society almost twenty millions to fight a disease that—at present death rates—will kill twenty-three million living Americans.

Less than one dollar for each American destined to die from cancer. Much more is needed for researeh, for education, for clinics. Won't you please do *your* part . . . now?

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enemy
strike back
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☐ Enclosed is my contribution of $..... to the cancer crusade.

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Simply address the envelope:
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Comparison of Several Methods and Instruments for Specifying the Color of Frozen Strawberries[a,b]

J. N. SHAH AND O. J. WORTHINGTON
Food Technology Department, Oregon State College

(Manuscript received June 13, 1953)

A comparative study of the validities and precisions of three instruments when used to estimate PMA color grades of Marshall and Northwest varieties is reported. The instruments are the Photovolt Reflectionmeter, the Hunter Color-Difference Meter and the Beckman DU Spectrophotometer used as an absorptometer. Also the best measurement for each instrument is determined. Two criteria are presented: the magnitude of the correlation coefficients of instrument readings and visual color scores and analysis of variance to determine the significance of the contributions of the several measures of a given instrument. No one instrument is shown to be universally more valid or precise than another; one's choice therefore being determined by costs and other factors. Correlation data between instruments are given.

In PMA grade standards for frozen strawberries, color accounts for 40% of the total score; it is therefore, without question, a critical factor in the total grade. Subjective estimates are used exclusively at present, since only one objective method for color of strawberries has been published *(7)*. This is a chemical method for anthocyanin concentration based upon optical density differences at two hydrogen ion concentrations. Therefore, the need for rapid physical (or psychophysical) methods is particularly urgent with this commodity. The Hunter Color-Difference meter, the Photovolt Reflectionmeter model 610 and the Beckman DU spectrophotometer used as an absorptometer were selected for the present study on the basis of their general usefulness and availability. Standardization of methods for these instruments, as applied to the color of frozen strawberries and their validities was considered first. These results led to model color specifications—using a representative but not exclusive criterion; namely, Hunter L—based upon several hundred samples of 10 varieties. The work of elucidation of more widely acceptable specifications should be carried on comprehensively—geographically and over several seasons—by a widely accepted organization in order that grade standards might be obtained on a definitive basis.

METHODS AND MATERIALS

Since most frozen berries originating in the Pacific Northwest are of the Marshall variety, these were the principal subjects for analysis for both 1951 and 1952 crops. However, the Northwest, a new variety, was also thoroughly investigated. It has a beautiful color and appearance and the flavor is comparable to that of Marshall.

In 1951 the berries were frozen at the laboratory at 10° F. in cellophane bags in 16-ounce cartons. In 1952, No. 1 cans were used. The berries were both frozen whole and sliced with sugar, the ratio being 3 + 1 (berries to sugar) in 1951, the more common (4 + 1) being used in the following year. The Marshalls were obtained rather late in the season the first year and the berries were not typical of the average pack. This fact was later indicated by the data.

All the packages of both varieties were numbered and the packages were withdrawn for examination with the aid of a table of random numbers.

In measuring the reflected color of strawberries there is a considerable problem in selecting the best method of preparation and presentation of the sample. For the fresh market, examination of the exteriors of whole berries may be sufficient, but most frozen strawberries are sliced and the interiors exposed. The color of the interiors is especially important in the case of frozen berries for the preserve trade. Data showed that the reproducibility was much better for pureed samples than for whole or sliced berries *(6)*. This would be expected since the variation among individual berries was eliminated. However, visual color grading could not be done on purees because their colors were so unfamiliar and unassociated with the usual appearance of strawberries that the judges were presented with a problem that was meaningless to them. Therefore, visual color grading was done first, followed by homogenization and determination of the purees from those berries. Since the judges were somewhat accustomed to a 1 to 10 scoring range, this scale was used. It is easily transformed to PMA color score points by multiplying by 4. The demarcations between the grades were the same as those of PMA. The scores of 6 judges who graded all the packages were included in the final results. In 1952, only one judge was used, since the results of the previous year showed that one of the experienced judges could be fairly substituted in place of the whole panel. One important reason for the change was the reduction in time required for scoring. A delay of one hour between time of thawing and time of instrumental color measurement permitted errors due to color changes.

The purees were made by blending in the Waring blender for 30 seconds followed by 15 minutes' vacuum deaeration. This was shown to introduce no appreciable color error.

The Hunter Color-Difference meter was standardized by means of the NBS red enamel plaque number SKC-51. The values in terms of %'s A, G, and B are provided with the plaque. These were converted to L, a_L, and b_L by using the formula given in the instruction manual. The large area opening was used. Three transparent plastic boxes, which gave the same Hunter readings, were used throughout the study as containers for the purees. Two hundred fifty g. of puree was sufficient. The L scale was used rather than Rd since the former showed a much larger range.

The Photovolt Reflection Meter was also standardized with the red enamel plaque No. SKC-51. The dark standard method as described in the instrument manual was used, because the percentage reflectances for all 3 tristimulus filters were very small. The error involved in reading the galvanometer is comparatively large, but this may be reduced by substituting a more sensitive galvanometer and increasing the resistance of the coarse control potentiometer as was done by Robinson *et al. (4)*. The search unit was placed in upright position and the sample in a plastic box placed on top of it.

Since the spectrophotometer without reflection attachment is commonly available, it was taken as the third instrument to be investigated. The Sondheimer and Kertesz method of quantitative determination of anthocyanin pigment in strawberries is available *(7)*, but a simpler and faster method of measuring color is needed. Moreover, a quantitative determination of pigment is

[a] Approved for publication as Technical Paper No. 815 with the approval of the Director of the Oregon Agricultural Experiment Station. Contribution of the Department of Food Technology.

[b] Presented at the Thirteenth Annual Meeting of the Institute of Food Technologists, Boston, Massachusetts, June 23, 1953.

unnecessary and not pertinent. From the preliminary experiments it was concluded that extracting the puree with a buffer at pH 3.40 ± 0.05 and measuring the optical-density at 500 mμ or taking a ratio at two wavelengths such as 500/375 mμ would give results which would correlate well with visual color and differentiate between color grades.

A 20-g. pureed sample was weighed into a micro-blender and 80 ml. of the citrate-hydrochloric acid buffer added. The mixture was blended for 15 seconds. After 15 minutes it was filtered through Whatman no. 5 paper. The Beckman DU spectrophotometer with 1 cm. quartz cuvettes were used. O. D. curves showed that the 2 varieties yielded similar curves with the major peaks at 500 mμ. The curves for B grade were similar to A but with lower O. D.'s throughout. The D grade curve was similarly lower than B grade. The Northwest variety has a higher O. D. at 500 mμ for both A and B grades than the Marshall variety.

In 1951, color determinations were made on 30 packages (unsweetened) of each of the 2 varieties by visual panel scoring, by the Hunter Color-Difference meter, and by the spectrophotometer. In 1952, color determinations were made on 60 packages of each of the same 2 varieties, half of the packages being sugared (4 + 1) and sliced. The methods utilized this year were visual, Hunter L and a_L/b_L, and Photovolt reflectionmeter % A and % G.

RESULTS AND DISCUSSION

Assessment of sources of color variance. Analysis of variance of the 1951 experiment showed among other things that the 3 color grades were readily distinguished by all 3 color estimation methods. Analysis of variance of the 1952 experiment verified the previous results; namely, that the different methods show a great deal of agreement. Further, there are shown in Table 1, and in Tables 2 and 3, the latter two of which are based on the best methods for each instrument, the effects of sugar, variety, and interactions. When berries were scored visually, no difference was found between those treated with sugar and those without sugar. Variety was not a factor in assignment of color scores.

The Hunter instrument (Table 2) distinguished differences between sugared and unsugared berries but no differences between the Hunter L values of berries of the same color grade but of different varieties. Analysis of variance of the Photovolt % G readings (Table 3) demonstrates the same sources of variance as the Hunter L readings. Likewise, the analysis of variance of the spectrophotometer optical density readings at 500 mμ in 1951 had essentially agreed with the former 2 instruments used in 1952.

Figure 1 shows the location of strawberry colors on the Hunter chromaticity graph to be fairly close to those of tomatoes (4). All workers have found that the ratio a_L/b_L gives a good demarcation between grades of tomato products. When this was applied to strawberries, the analysis of variance gave the same results as with L readings except that the F values were lower. This is one bit of evidence for preferring the L scale; in addition, it is simpler. All of these variance statistics point to the desirability of investigating the correlations of the various instrument readings with visual PMA type scores.

TABLE 1
Analysis of variance of visual scores for 120 packages[a] of Marshall and Northwest berries in 1952

Variation due to:	Sum of squares	Degrees of freedom	Mean squares	F
Sugar	0.2083	1	0.208	0.11
Grade	2127.6500	2	1013.825	506.91**
Variety	1.0083	1	1.008	0.53
Sugar x Grade	1.0167	2	0.508	0.27
Grade x Variety	14.5167	2	7.258	3.85*
Sugar x Variety	0.4084	1	0.408	0.21
Sugar x Grade x Variety	2.8166	2	1.408	0.75
Error	203.7000	108	1.886	
Total	2351.3250	119		

[a] 60 of these were whole, unsugared, and 60 were sliced and sugared (4 + 1).
* Significant at 5% level.
** Significant at 1% level.

TABLE 2
Analysis of variance of Hunter L for 120 packages[a] of Marshall and Northwest berries in 1952

Variation due to:	Sum of squares	Degrees of freedom	Mean squares	F
Sugar	3221.4413	1	321.441	64.62**
Grade	504.1755	2	252.088	50.68**
Variety	1.1213	1	1.171	1.97
Sugar x Grade	9.9482	2	4.974	8.32*
Sugar x Variety	0.5470	1	0.547	0.92
Grade x Variety	3.8802	2	1.940	3.28
Sugar x Grade x Variety	0.9995	2	0.500	0.84
Error	64.2140	108	0.594	
Total	905.8370	119		

[a] 60 of these were whole, unsugared, and 60 were sliced and sugared (4 + 1).
* Significant at 5% level.
** Significant at 1% level.

TABLE 3
Analysis of variance of Photovolt % G for 120 packages[a] of Marshall and Northwest berries in 1952

Variation due to:	Sum of squares	Degrees of freedom	Mean squares	F
Sugar	51.3128	1	51.313	48.55**
Grade	75.4661	2	37.733	35.70**
Variety	0.4928	1	0.493	3.03
Sugar x Grade	2.1137	2	1.057	6.50*
Sugar x Variety	0.3934	1	0.393	2.42
Grade x Variety	0.4305	2	0.215	1.32
Sugar x Grade x Variety	0.2430	2	0.121	0.74
Error	17.5500	108	0.162	
Total	148.0013	119		

[a] 60 of these were whole, unsugared, and 60 were sliced and sugared (4 + 1).
* Significant at 5% level.
** Significant at 1% level.

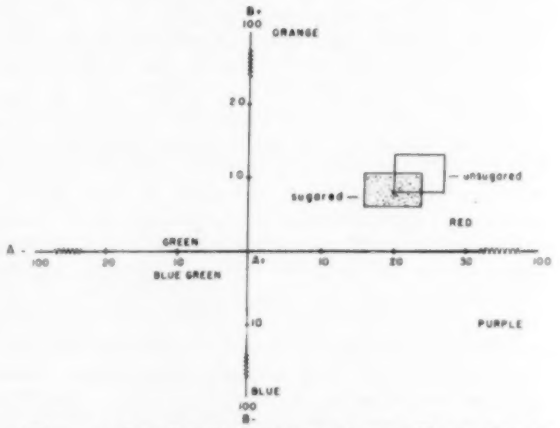

Figure 1. Location of strawberry colors on Hunter chromaticity graph.

Validity of the instrumental methods. Multiple correlation coefficients for each variety and each treatment

were calculated separately (Table 4). In the case of whole berries without sugar, the R^2 values for both varieties were very close. The analysis of variance results having shown no difference between the two varieties, the readings were pooled, Table 4, line 4. Simple correlations between Hunter L and visual scores were also calculated because the data of the previous year indicated that the use of L alone was sufficient. The r^2 values presented in Table 4 indicate that simple correlations using L alone are approximately as good as

TABLE 4
Comparison of correlations between visual scores and various instrumental methods

Treatment and variety	No. of samples	Instrument	*1952* R^2 or r^2	*1951* R^2 or r^2
1. Northwest whole	30	Hunter L, a_L, b_L	0.880	.888
Northwest whole	30	L	0.830	.878
2. Marshall whole	30	Hunter L, a_L, b_L	0.870	.867
Marshall whole	30	L	0.838	.854
3. Marshall sliced and sugared	30	Hunter L, a_L, b_L	0.762	
Marshall sliced and sugared	30	L	0.686	
4. Marshall and Northwest whole	60	Hunter L, a_L, b_L	0.862	
		L	0.833	
5. Marshall whole	30	Photovolt:		
		% A and G	0.808	
		% A	0.780	
		% G	0.804	
		% A, G, B	0.818	
6. Marshall sliced and sugared	30	% A	0.704	
		% G	0.693	
7. Marshall whole	30	500 mg		0.743
Marshall whole	30	Ratio 500/375		0.883
8. Marshall sliced and sugared	20	500 mg		0.819

correlations using all 3 Hunter scales. Likewise with the Photovolt reflectionmeter, % G is practically as valid as using all 3 measures, as evidenced by correlation coefficients. These results are a verification of some previous work with the Photovolt *(5)*. Thus the instrumental methods of Table 4 are all valid measures of the visual color score.

Multiple vs. simple regression. If Hunter L reading alone (or Photovolt % G) can be used without loss of accuracy due to discarding the other 2 readings it would be most desirable to do so. In specifying color standards and representing them in graphical form, 3-dimensional graphs become complicated and difficult to visualize. Therefore, analysis of variance of the visual color scores was made (*3*, p. 133) to determine the contribution of the factors other than L to the multiple regression equation, as shown in Table 5, lines 1, 2, and 3. The contributions of a_L and b_L are found not to be significant, a verification of the conclusion drawn from inspection of correlation coefficients. Lines 4 and 5 of Table 5 show that b_L cannot be used alone since L and a_L make significant contributions to the regression coefficients. In the case of % G, its simple correlation coefficient was so close to the multiple correlation coefficient that verification by analysis of variance is unnecessary. In case of

TABLE 5
Analysis of variance of visual color scores for 30 packages of Marshalls, 1951

Source of variation	D.F.	s.s.	m.s.	F
Regression due to:				
1. L, a_L and b_L	3	901.20	300.4	5.7*
2. L *Alone*	1	887.63	887.6	167. **
3. a_L and b_L (adjust. for L)	2	13.56	6.8	1.3
4. b_L *Alone*	1	707.84	707.8	133. **
5. L and a_L (adjust. for b_L)	2	193.36	96.7	18.2**
6. *Residual*	26	138.17	5.3	
7. Total	29	1039.37		

* Significant at 5% level.
** Significant at 1% level.

doubt concerning R^2 and r^2, analysis of variance will resolve the issue.

Construction of model color specifications. Validity of instrumental color determination in the case of strawberries means that the instrumental methods should agree with visual grading in terms of PMA color scores, unless and until one wishes to undertake to show that PMA color scores are "wrong" from the consumers' standpoint. Since validity has been shown for the best measure for each instrument, the next step in putting the conclusions into practice is to know the regression equations for predicting PMA grades from the instruments. These data are shown in Table 6. Also the standard errors of estimate in terms of the 40-point PMA scale are given.

Because of the facts above and because of the sensitivity of the Hunter L scale, it was further studied as an example of the setting up of a practical regression line for predicting PMA scores. The standard error in Table 6 is fairly large. Much of that error is due to the unreliability of subjective grading. It was decided to reduce the error by classifying the readings from a large number of packages into classes covering a range of 0.5L unit for each. One hundred forty-one packages of 5 varieties, sliced and sugared (4 + 1) are shown in the scatter diagram and regression line of Figure 2. The averages of the visual scores of each classification are plotted against the average L of that group. Thus, the standard error is reduced to ±1.70 PMA units when

TABLE 6
Regression equations of selected methods for 1952

Variety	Instrument	r^2	$Sy.x$ †	Regression equations
Marshall, unsugared	Hunter L	0.84	1.99	PMA = 71.69 − 1.738L
Marshall, unsugared	Photovolt % G	0.80	2.19	PMA = 56.78 − 4.175% G
Northwest, unsugared	Hunter L	0.83	1.75	PMA = 68.37 − 1.600L
Northwest, unsugared	Photovolt % G	0.79	1.93	PMA = 53.66 − 3.806% G
Northwest, sugared	Hunter L	0.83	1.805	PMA = 75.22 − 2.238L
Northwest, sugared	Photovolt % G	0.72	2.34	PMA = 54.55 − 4.910% G
Marshall, sugared	Hunter L	0.68	2.631	PMA = 66.47 − 1.748L
Marshall, sugared	Photovolt % G	0.69	2.60	PMA = 52.76 − 4.586% G

† In terms of PMA color score points.

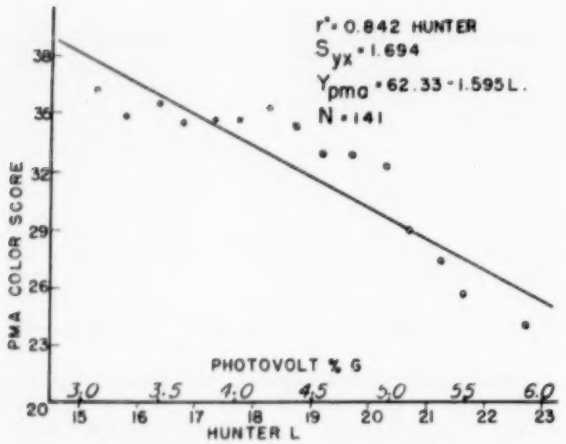

Figure 2. Regression line for estimating PMA color scores.

5 or 6 packages are averaged in making an estimation, even though 5 varieties were used. Thus the regression diagram or equation is now usable with known precision.

As stated earlier, a separate equation is desirable for unsugared berries in the case of Hunter L readings. When 221 packages of unsweetened berries of 10 varieties were classified and plotted in the same way, r^2 was calculated to be 0.936 and the $S_{y.x}$ was $\pm$ 1.39 PMA units. In this case, PMA score equals 61.47 - 1.362L.

The use of Hunter L serves as a demonstration or model. A regression equation could likewise be calculated for Photovolt % G, especially with data from berries of a particular production or variety, using the average reading from several replicates. The % G scale in Figure 2 has been added from the relationship between % G and Hunter L shown in Figure 3.

Correlation between instruments. If it is desired to measure PMA color scores without accumulating data from one's own production, this can be done by substituting some other instrumental measure for Hunter L if the two show high correlation. Table 7 shows correlation coefficients between selected instruments. Figure 3 shows the very good correlation of Photovolt % G with the Hunter L. The former can then be substituted with confidence, where Hunter L has previously been mentioned, such as in Figure 2, using the regression equation to convert the L scale to a % G scale.

Converting instrumental results into standard systems of color specification. It is standard practice in most color measurement work to express final results in terms of standard color systems, either physical or psychophysical. The primary reason for doing this is to provide a common basis for interpreting and comparing data, thus permitting greater advance in the field of color specification. In the earlier literature, most color data were expressed in terms of dominant wavelength, brightness and purity. In 1931, due to the effort of workers in this field, the International Commission on Illumination (I. C. I.) set up psychophysical standards. The Munsell color system also has been widely used in recent years *(8)* to define and describe the color of a large variety of products. (See Judd, *2*, p. 224).

TABLE 7
Correlation between various instrumental methods [a]

Variety	Instrument	r^2	N
Northwest—whole	Hunter L vs. O. D. at 500	0.91	30
	Hunter L vs. Ratio 500/375	0.68	30
Marshall—whole	Hunter L vs. Ratio 500/375	0.90	30
Both varieties—whole	Hunter L vs. % G	0.94	60

[a] Pureed, unsugared.

The results of *2* instrumental methods on 10 packages of strawberries representing the total color range were converted to I. C. I. and dominant wavelength in order to determine whether the *2* instruments give the same converted results (Table 8). Theoretically, the instruments used are capable of giving identical results when converted into I. C. I., dominant wave length or Munsell systems. The table clearly shows that in actual practice this is not true. The values obtained from the *2* instruments are quite far apart even though they show a similar trend.

Some other workers in this field also have compared the results of two or more instruments after converting to a standard color system. Robinson *et al. (4)* measured tomato juice color using both the Hunter Color-Difference meter and the Beckman spectrophotometer on the same cans of juice. In their Table 1, the results converted to dominant wave length show considerable differences between the two instruments. In their Table *2*, color measurements with the Hunter and Photovolt for the same can of juice are presented. If we convert the results into terms of I. C. I., a great difference in values is found.

Friedman, Marsh and Mackinney *(1)* also compared the results obtained from the G. E. Recording and the Beckman Du Spectrophotometers (the same instru-

TABLE 8
Conversion of instrumental results to I. C. I. and dominant wave length

Sample No.	From Hunter				From Photovolt			
	I. C. I.			Dominant wave length	I. C. I.			Dominant wave length
	Y%	x	y		Y%	x	y	
73	2.37	0.492	0.307	627	3.25	0.4555	0.3403	601
61	2.86	0.536	0.321	614	4.00	0.4987	0.3358	605
80	3.24	0.535	0.325	612	4.25	0.4958	0.3242	611
33	3.72	0.531	0.337	606	5.00	0.5026	0.3785	593
2	3.92	0.554	0.327	611	4.87	0.5871	0.3642	600
35	4.58	0.526	0.329	609	5.00	0.5127	0.3499	600
23	5.02	0.529	0.331	608	5.87	0.5281	0.3626	598
18	5.52	0.531	0.334	607	5.62	0.5265	0.3506	601
40	6.10	0.519	0.336	605	5.24	0.5017	0.3529	599
55	7.02	0.495	0.346	601	7.60	0.4913	0.3664	595

ments, in principle) and found a difference between identical samples of 2 mμ in terms of dominant wave length.

From the discussion above it would seem that one of the apparent advantages of reporting the data in standard systems is lost, since different instruments give different values for the same sample. It must also be pointed out that the conversion of the results of different instruments to a standard system is quite involved and requires a fairly good understanding of the theoretical background of the subject. In some cases, the converted results may lead to wrong interpretations and erroneous conclusions if one is not aware of the errors involved. With strawberries, our results show that there is no need of converting into terms of a standard system, for the measurements by different instruments may be compared on the basis of regression lines as illustrated in Figure 3 and as applied in Figure 2.

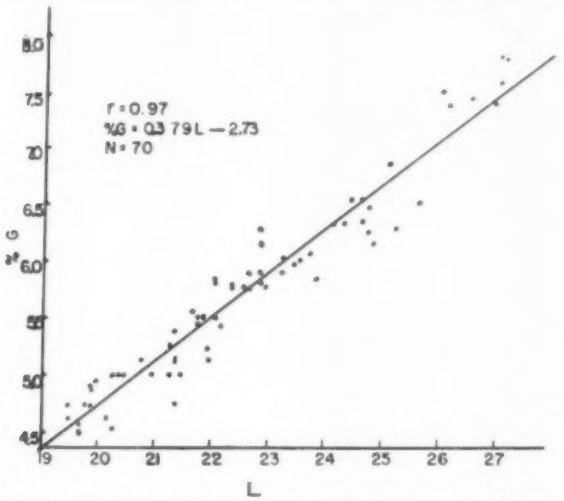

Figure 3. Correlation of best single measures of two reflectionmeters.

These comments pertain to procedures which may be adopted at once for quality control and similar purposes. On the other hand, discrepancies in converted data must be studied further and reduced in order to establish absolute standards not linked to any specific make of instrument.

SUMMARY AND CONCLUSIONS

Ninety samples of frozen strawberries of each of two varieties, over two seasons, were analyzed for color by two or three measures for each of three instruments. The Marshall variety is reported principally. The Northwest variety is also analyzed, as well as a few data from experimental varieties. Analysis of variance shows the factors having a significant effect on visually or instrumentally measured color. With the latter, the addition of sugar to sliced berries to produce a (4 + 1) pack, produced a significant effect on the color. Therefore, different instrumental specifications are required for sugared (4 + 1) berries than for whole unsugared berries.

The validity of the many methods is compared by the magnitudes of their correlation coefficients with PMA type scores. This is one way of deciding whether multiple correlations, using all three measures for the Hunter or the Photovolt reflectionmeters are necessary. These conclusions can be verified in doubtful cases as illustrated by analysis of variance of the visual color scores as affected by instrument readings. It was found that Hunter L is practically as valid as all three measures of that instrument. Likewise Photovolt % G is satisfactory alone.

Regression data for the best single measures for each of the reflection meters are given. This includes complete regression equations and the standard errors of estimate. Thus the effectiveness of the several methods in estimating visual scores is apparent. Pooled data of 141 samples of 5 varieties, sliced (4 + 1), yield results which may be considered reliable enough to use now until data more specific for one's own purposes can be accumulated.

Correlation coefficients between instruments show the possibilities of substituting one instrument for another. One instrument is not irrefutably better than another, but one's choice may be determined by many factors other than validity so long as the best measure for each instrument is utilized.

Conversion of the instrumental data to standard color systems such as ICI is shown to involve large errors and to be unnecessary for quality control purposes.

Not reported here are the experiments which showed quantitatively the best methods for preparation of the samples and presentation to the instruments, nor data comparing the anthocyanin pigment determination method with other rapid absorptometer methods.

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A Critical Examination of Objective Methods for Maturity Assessment in Frozen Peas[a]

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Objective methods for determining maturity in frozen peas are critically examined to determine their comparative reliability. Present methods are shown to be quite satisfactory except for samples in the borderline zone. The methods of specific gravity and of alcohol-insoluble solids are the most reliable of the objective tests proposed thus far. It is suggested that these methods plus the brine flotation test could be used for borderline zone samples.

The pea is a vegetable of great economic importance and, as far as the frozen pack is concerned, more poundage of it is packed than any other frozen vegetable *(21)*. In view of this fact, a thorough elucidation of the determination of quality in frozen peas is desirable. Investigation was centered around maturity at harvest since this is assumed to be the dominant factor in quality, and work was undertaken on a number of objective methods for the assessment of maturity.

Three different types of methods exist at the present time for the evaluation of the maturity of peas; namely, mechanical, physical, and chemical. The methods for raw peas involve the use of mechanical instruments, of which the tenderometer *(14)* and the maturometer *(12)* are examples. The methods for processed peas are based on physical and chemical techniques. Examples of the physical methods are those involving the use of specific gravity, two of which have been suggested *(8, 9, 22)*. Chemical methods are based on the determination of alcohol-insolube solids *(4, 5)*, and starch *(1, 18, 20)*.

REVIEW OF THE LITERATURE

Among the papers published on this subject, the following are of particular interest. In 1934 and 1935, Kertesz *(4, 5)* proposed the use of the determination of alcohol-insoluble solids as a means for the evaluation of the maturity of canned peas. In 1938, Martin, Lueck, and Sallee *(14)* described the use of the tenderometer, an instrument which can be used for the determination of the relative maturity of fresh peas by recording the force necessary to shear them through a standard grid. In 1941, Lee *(8, 9)* proposed a test to measure the maturity of frozen peas based on differences in specific gravity. This test was modified in 1942 *(10)*. In 1943 Nielsen *(18)* suggested a test based on the determination of the starch content of frozen peas by solubilizing it with perchloric acid followed by measurement of the color formed by iodine. This test was modified in 1945 by Nielsen and Gleason *(20)*. Tentative United States Standards for Grades of Frozen Peas, effective March 15, 1945 *(22)* include a test for tenderness and maturity based on the buoyancy of skinned peas in a series of standard salt solutions. This has been known as the brine flotation test. In 1947, Nielsen, Campbell, Bohart, and Masure *(19)* discussed several of the methods used for the assessment of the maturity of frozen peas. In that paper, they stressed the methods based on the determination of starch content and of total solids content as objective indices to be used for the measurement of the maturity of frozen peas.

In 1948, Moyer and Holgate *(17)* published a modification of the method of alcohol-insoluble solids. Lynch and Mitchell, 1950 *(12)* studied the use of the determination of alcohol-insoluble solids, and of the specific gravity method, together with the tenderometer and maturometer for the evaluation of the maturity of canned peas and raw peas for canning. Recently (1953) Makower, Boggs, Burr, and Olcott *(15)* published a comparison of methods for measuring the maturity factor in frozen peas. The conclusions arrived at in that article indicate that alcohol-insoluble solids, cotyledon texture, and skin texture showed good reliability as indices of maturity for frozen peas. According to them, specific gravity and the percentage of total solids correlated moderately well with subjective tests. A test yielding an index of the amylose content of the peas showed a varietal difference in its relationship to subjective scores. For this reason, it was abandoned.

MATERIALS AND METHODS

Six varieties of peas, Thomas Laxton, Tall Telephone, Wyola, Shasta, Deep Green Perfection, and Onward, most of which have been in commercial use in this area, were employed. These are all the wrinkled skinned type of peas normally used in the freezing industry. They were grown on the Station farms, so that a minimum of delay would result between harvest and processing. Because of a crop failure in 1947, however, the peas were obtained from a point about 20 miles from the Station, by special arrangement. All precautions were taken to transport them to the laboratories with a minimum of delay. It is the opinion of the workers in this laboratory that peas to be used for maturity studies should be grown near the laboratories in which the tests are to be made, if wholly satisfactory results are to be obtained.

Each variety was obtained at several different stages of maturity. The bulk of them, however, were in the *fancy* and *extra standard* range. In accordance with standard practice in the industry, the peas were ungraded in all these tests. They were vined, using a commercial viner available on the Station grounds, and cleaned, during which operation the unusable peas, smaller than size grade no. 1, were removed. Immediately after this, tenderometer readings were taken. Each lot was blanched without delay in boiling water for 60 seconds, cooled in cold water, and drained. They were then passed over a picking table for removal of foreign matter, halves, and skins. The peas were then packed in waxed cups[b]—especially lined with moisture-vapor-proof cellophane bags, frozen, and stored at 0° F. (−17.8° C.).

After blanching, the peas were purposely not brine separated, so that the tenderometer readings on the raw material could be correlated with the indices of maturity run on the corresponding processed samples.

The results reported in this paper are limited to those obtained with the 1949 crop. Similar results were obtained for the other 2 years; hence it was considered unnecessary to report them here. All determinations were carried out in duplicate, unless otherwise indicated.

Tenderometer Readings for Raw Peas. Tenderometer readings were taken in the neighborhood of 75° F. (23.9° C.).

The following tests were run on the precessed samples:

Brine Flotation Test. The recommendations described in the U. S. Standards for Grades of Frozen Peas *(22)* were employed. Room temperatures were used, as directed, and in all cases the peas and the brines used were at the same temperature.

[a] Approved for publication by the Director of the New York State Agricultural Experiment Station as Journal Paper No. 932.

[b] Kindly supplied by Lily Tulip Cup Corporation, New York, N. Y.

Specific Gravity. This determination was made in 2 ways. First, the thawed peas were plunged into boiling distilled water and cooked for 5 minutes after resumption of boiling. They were then cooled under water to 20° C. (68° F.), drained, and, following this, a 100-g. sample was weighed in air, and again weighed submerged in water at 20° C. (68° F.). Before weighing the second time, bubbles of air were removed by carefully stirring with a glass rod. This procedure should be completed in a minimum of time.

The second method differed from the first in that each pea was punctured by means of the instrument, fitted with darning needles shown in Figure 1, prior to boiling for 5 minutes, and cooling under water as described before.

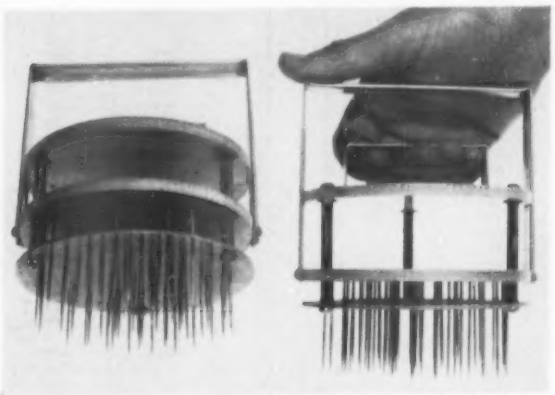

Figure 1. Device for puncturing peas.

Alcohol-Insoluble Solids. This index was run according to the method of Moyer and Holgate *(17)*. The 20-g. sample was blended by whipping in a Waring blender with 85% ethyl alcohol, filtered on tared filter paper, dried over night at 95° C. (203° F.), cooled, and weighed.

Total Solids. Total solids were run on 20-g. samples of peas. The samples were first taken to near dryness on the steam bath, transferred to the vacuum oven, and dried at 70° C. (158° F.) for 48 hours.

Starch. This substance was determined by the Nielsen method *(18, 20)* starting with a 100-g. sample. The standard starch employed in this work was prepared from each of the varieties under consideration for use in the determination of starch in that particular variety. Shasta was the only variety for which this was not done. In the determination of starch in this variety, Thomas Laxton starch was used as the standard. Peas of a tenderometer reading greater than 160 were used for the preparation of the starch. Special precautions were taken to ensure recovery of starch in pure form. The pea slurry, obtained as suggested by Nielsen *(18)*, was strained through a 100-mesh screen, and the starch was separated by a very large number of decantations with distilled water. This was done by pouring very rapidly from full jar to empty jar, and combining the small amounts of sedimented starch remaining behind. In the final washings with alcohol, no color was removed by this solvent, and in the finished product no foreign particles could be found when examined under the microscope.

In the 1949 series, starch was run on the dried peas from which sugars had been removed by extraction in the Soxhlet apparatus with 80% ethyl alcohol, by a modification of the diastase method with subsequent acid hydrolysis, recommended by the A. O. A. C. *(1)*. According to Kurgatnikov *(6)* hydrolysis with diastase is difficult in the case of starches prepared from wrinkled peas. In an attempt to overcome this difficulty the pea sample in each case was autoclaved for 15 minutes at 15 pounds of steam pressure prior to treatment with diastase. In this way, the starch particles apparently disappeared after 3 to 4 treatments with diastase. These data are slightly higher than those obtained by the other method, and are not presented in Table 1 because the method is too involved to be of much value.

Organoleptic tests. Approximately 2 cups of frozen peas were added to ¾ cup of boiling water and cooked for 8 minutes after resumption of boiling. They were drained and served to the tasters without seasoning.

Prior to the regular tests the tasters were given a preliminary test with the following grades of Thomas Laxton frozen peas: *fancy, extra standard, standard, and substandard.*

A score sheet was used with a 1 to 10 scale. One on this scale was *extremely poor*, 9 *very good*, and 10 *excellent*. In practice this was found to approximate a 9-point scale because rarely was a sample given a rating of 10. The peas were scored for texture by a selected panel of 14 tasters. In order that each taster would have only 4 samples to compare at one time instead of 8, the following balanced incomplete block design (plan 11.10, page 330, reference *3*) was used.

Groups of 8 samples were chosen with ranges in tenderometer readings from low to intermediate and high and in one case, low to high. The samples in the groups with a range in tenderometer readings were randomized and each taster had a range of maturities from low to intermedite or low to intermediate and high, as indicated by the tenderometer readings.

This system might work out so that one taster of the group might have a range of low tenderometer readings and one person a range of high readings, but in most cases the tasters would have a satisfactory range for comparison. The blocks used are as follows:

	Rep. 1	*Rep. 2*	*Rep. 3*	*Rep. 4*
Block	(1) 1 2 3 4	(3) 1 2 7 8	(5) 1 3 6 8	(7) 1 4 6 7
	(2) 5 6 7 8	(4) 3 4 5 6	(6) 2 4 5 7	(8) 2 3 5 8

Rep. 5	*Rep. 6*	*Rep. 7*
(9) 1 2 5 6	(11) 1 3 5 7	(13) 1 4 5 8
(10) 3 4 7 8	(12) 2 4 6 8	(14) 2 3 6 7

Each day, *fancy, extra standard, standard,* and *substandard* samples from the same standard lots were used by the tasters for comparison.

The average deviation from the mean score for 12 of the 14 tasters was less than one, ranging from 0.62 to 0.95, with 2 tasters having average deviations of 1.14 and 1.17.

RESULTS

The results of this investigation into the comparative practical application of objective methods for the determination of maturity of peas are set out in Tables 1 and 2, and in Figures 2-8.

Table 3 gives the results of a replicate study of reproducibility of values for specific gravity and for starch determined by the Nielsen method.

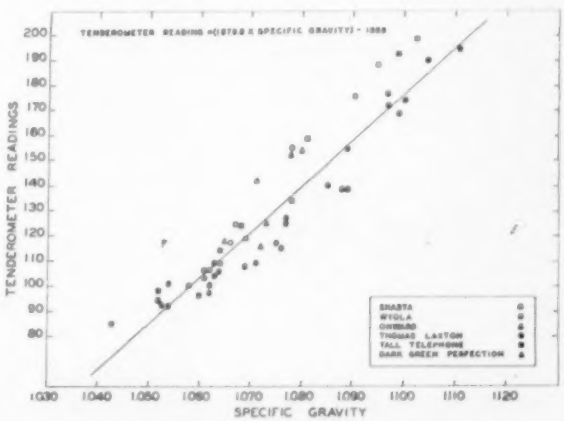

Figure 2. Comparison of tenderometer readings with specific gravity.

TABLE 1
Pea maturity study
1949

Sample No.	Taste	Tenderometer	Brine flotation test	Sp. Gr. cooked only	Sp. Gr. punctured and cooked	Total solids	Alcohol-insoluble solids	Starch (Nielsen)
Thomas Laxton						*Percent*	*Percent*	*Percent*
4198	8.7	94	38	1.048	1.052	19.1	11.7	3.50
4199	8.5	92	38	1.050	1.053	18.4	11.9	3.78
4200	8.1	101	35	1.057	1.054	20.7	13.0	4.28
4201	8.2	96	37	1.054	1.060	20.8	12.6	4.14
4202	8.3	97	34	1.062	1.062	21.2	13.3	4.51
4203	8.0	100	38	1.055	1.062	20.1	13.5	4.28
4204	6.5	108	34	1.067	1.069	22.7	16.0	5.25
4205	6.9	106	34	1.067	1.064	21.7	13.1	4.80
4206	9.3	92	38	1.049	1.053	18.1	10.7	3.18
4207	8.4	104	35	1.062	1.063	21.1	13.3	5.41
4208	7.1	109	33	1.067	1.071	21.7	14.1	5.07
4210	6.7	117	28	1.071	1.075	22.6	15.5	5.35
4211	7.5	115	29	1.076	1.076	23.6	13.1	5.81
4212	5.7	127	27	1.080	1.077	23.6	16.8	5.58
4213	7.7	125	29	1.076	1.077	22.8	16.2	5.65
4221	8.3	109	35	1.067	1.063	19.7	16.7	5.82
4225	4.7	155	—	1.088	1.089	26.3	19.9	7.48
4227	3.9	172	—	1.094	1.097	28.1	22.5	8.90
4228	3.0	177	—	1.093	1.097	27.8	22.3	9.06
4222	5.3	140	—	1.085	1.085	25.7	20.3	7.05
4223	5.4	139	—	1.079	1.088	24.7	17.7	6.13
4224	4.6	139	—	1.086	1.089	25.2	18.5	6.22
4229	3.0	190	—	1.100	1.106	30.0	23.4	8.32
4230	3.4	174	—	1.100	1.100	28.3	22.6	8.89
4231	3.7	169	—	1.098	1.099	27.9	22.1	8.86
4238	1.8	195	—	1.112	1.112	30.0	25.2	10.08
Wyola								
4214	8.0	103	39	1.062	1.061	20.9	11.3	3.44
4216	8.0	106	37	1.061	1.062	20.1	11.0	3.59
4217	6.4	125	35	1.066	1.067	22.4	13.0	3.94
4218	8.7	100	38	1.058	1.058	20.7	11.0	3.66
4219	8.4	106	35	1.061	1.061	21.5	11.7	4.04
4220	8.3	109	36	1.064	1.064	20.4	11.3	3.51
4233	8.4	85	40	1.041	1.043	25.2	9.1	2.23
4234	6.4	134	31	1.078	1.078	23.1	15.2	5.25
4235	6.8	119	37	1.068	1.069	20.5	13.3	4.74
4239	2.4	198	—	1.101	1.103	30.6	24.7	9.43
4242	5.1	159	—	1.082	1.081	24.4	17.3	6.31
4248	3.1	189	—	1.092	1.094	27.8	21.9	7.75
Onward								
4241	7.3	118	—	1.065	1.066	20.7	13.7	4.39
4247	4.7	142	27	1.067	1.071	22.2	16.2	5.75
4255	4.7	154	27	1.079	1.080	24.8	18.5	5.88
Tall Telephone								
4240	8.6	98	33	1.050	1.052	17.2	10.1	3.20
4245	6.4	114	33	1.063	1.064	19.6	13.2	4.48
4254	6.1	124	30	1.068	1.068	21.4	14.9	5.20
4256	2.9	193	—	1.097	1.099	28.9	23.8	9.70
Shasta								
4243	5.3	155	29	1.078	1.078	23.5	16.5	5.63
4236	6.4	117	35	1.063	1.066	19.7	12.9	4.03
4246	4.4	176	—	1.089	1.090	25.5	19.3	6.26
Deep Green Perfection								
4237	7.4	116	34	1.070	1.072	20.1	12.8	4.34
4244	6.7	125	31	1.067	1.073	20.3	13.0	4.78
4249	4.9	153	—	1.076	1.078	23.6	17.2	6.56

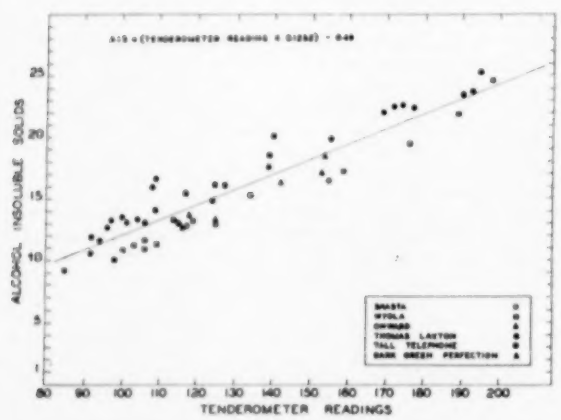

Figure 3. Comparison of alcohol-insoluble solids and tenderometer readings.

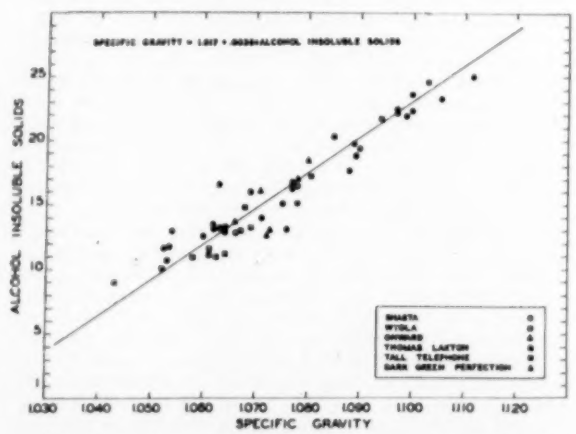

Figure 4. Comparison of alcohol-insoluble solids and specific gravity.

TABLE 2
Statistical relationships
(1949 series)

Relationship	Maturity range	Pairs of observations	Coefficient of correlation (r)	Standard error of estimate (S_{ya})
Specific gravity (boiled only) and tenderometer	All Stages	50	+0.95 ± 0.015**	0.0053
	Restricted*	31	+0.87 ± 0.043	0.0044
Specific gravity (punctured and boiled) and tenderometer	All Stages	50	+0.95 ± 0.014	0.0052
	Restricted	30	+0.87 ± 0.044	0.0040
Specific gravity (punctured and boiled) and alcohol-insoluble solids	All Stages	51	+0.95 ± 0.013	0.0050
	Restricted	31	+0.74 ± 0.083	0.0057
Alcohol-insoluble solids and tenderometer	All Stages	50	+0.94 ± 0.015	1.43
	Restricted	31	+0.64 ± 0.105	1.47
Total solids and tenderometer	All Stages	50	+0.92 ± 0.023	
	Restricted	31	+0.40 ± 0.151	1.41
Brine flotation test and tenderometer	Down to and including the samples just given the Federal grade of 27.	34	−0.80 ± 0.062	2.32
Starch (Nielsen) and tenderometer	All Stages	50	+0.92 ± 0.022	0.78
	Restricted	31	+0.65 ± 0.104	0.69
Starch (diastase method with subsequent acid hydrolysis) and tenderometer	All Stages	50	+0.96 ± 0.012	
	Restricted	31	+0.65 ± 0.103	0.45
Specific gravity (boiled only) and taste test	All Stages	50	−0.94 ± 0.017	0.0057
	Restricted	31	−0.69 ± 0.093	0.0064
Specific gravity (punctured) and taste test	All Stages	50	−0.94 ± 0.015	0.0054
	Restricted	30	−0.69 ± 0.096	0.0059
Tenderometer and taste test	All Stages	50	−0.97 ± 0.010	0.53
	Restricted	31	−0.81 ± 0.063	0.56
Alcohol-insoluble solids and taste test	All Stages	50	−0.94 ± 0.016	1.44
	Restricted	31	−0.60 ± 0.115	1.53
Starch (Nielsen) and taste test	All Stages	50	−0.92 ± 0.023	1.02
	Restricted	31	−0.53 ± 0.128	0.76

* Up to and including a tenderometer reading of 128.
** Standard error.

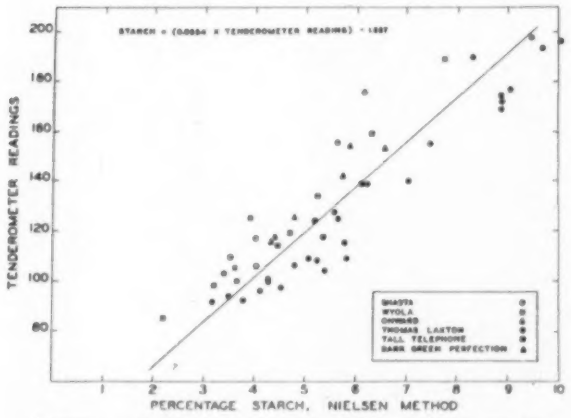

Figure 5. Comparison of tenderometer readings and percentage starch, Nielsen method.

DISCUSSION

Raw peas. The only mechanical device tested in this series of experiments was the tenderometer. When the figures obtained by the use of this instrument were correlated with the organoleptic tests, a very high correlation was obtained. Since any test to be used for the evaluation of the maturity of a sample must give good correlation with the results obtained by a highly qualified taste panel, the tenderometer under the conditions used in this work can be considered to give satisfactory performance when the peas have been properly sampled before testing *(7)*. The question arises in this connection of strict maintenance of standard operational conditions. Temperature has been found to be a factor in the use of the tenderometer, and since it was suggested *(2)* that a difference of 1° F. varies the reading by approximately 0.25, and in view of the fact that tenderometer readings are good to about 2 points on the scale *(7)*, it would appear that relatively large temperature differences are necessary to have an effect on the readings that is of practical significance. Such differences are encountered when readings are made on lots of peas which have been immersed in ice water and compared with readings made on the same material held at room temperature. While extreme precautions in the use of this instrument are of questionable value, moderate precautions are in order. This view appears to be confirmed by the observations of Martin, Lueck, and Sallee *(11)* who state "The curves . . . show that the

TABLE 3
Replicate study, Thomas Laxton peas
(Tenderometer reading of sample: 92)

No.	Sp. Gr. cooked	Sp. Gr. punctured	Starch Nielsen
1	1.049	1.054	3.58
2	1.048	1.053	3.55
3	1.048	1.052	3.38
4	1.048	1.053	3.00
5	1.048	1.053	3.45
6	1.048	1.053	3.00
7	1.049	1.054	3.38
8	1.049	1.052	3.55
9	1.050	1.056	3.45
10	1.050	1.056	3.38
11	1.048	1.055	3.80
12	1.048	1.055	3.38
13	1.049	1.056	3.52
14	1.048	1.055	3.82
15	1.048	1.055	3.90
16	1.048	1.055	3.82
17	1.050	1.052	3.60
18	1.048	1.054	3.55
19	1.050	1.055	3.82
20	1.049	1.056	3.62
	Av. 1.049	Av. 1.054	Av. 3.53

In each case, 100 g. of fresh material was used so that each was a determination on separate samples.

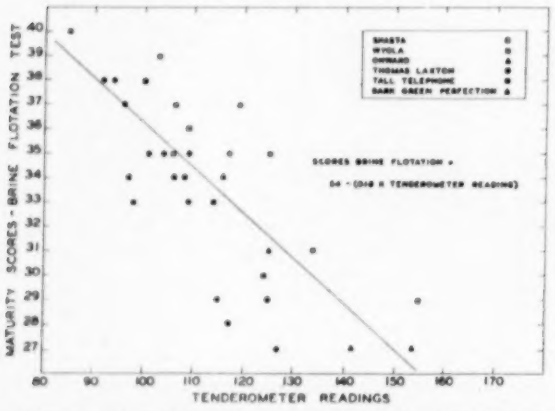

Figure 6. Comparison of maturity scores-brine flotation test and tenderometer readings.

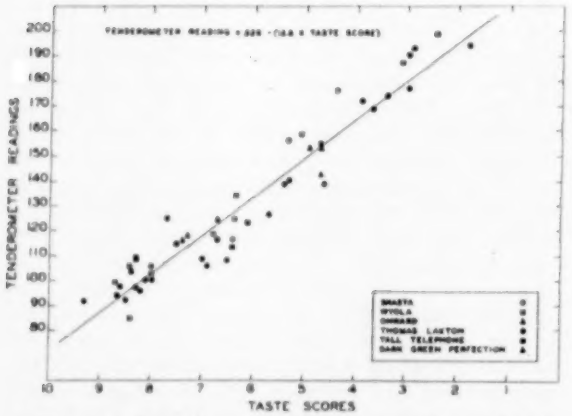

Figure 7. Comparison of tenderometer readings and taste scores.

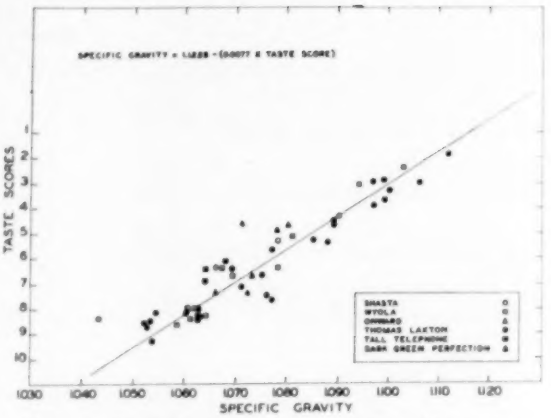

Figure 8. Comparison of taste scores and specific gravity.

tenderometer readings for both raw and canned peas decreased significantly with increasing temperature, hence in cases of extreme temperature variations, a correction should perhaps be applied."

In the series of experiments described in this paper the pea samples were tested by the tenderometer when the temperature approximated 75° F. (23.9° C.), but for comparative purposes any other selected temperature could have been used as a matter of convenience.

Lynch and Mitchell *(13)* found a tenderizing effect from increasing viner speed on comparable samples of peas. If this observation is confirmed, the need for strict standardization of the viner speed is indicated to obtain accurate results.

Processed peas. The brine flotation test is the standard procedure used by the U. S. Department of Agriculture *(22)* to determine the maturity and tenderness of frozen peas. Removal of the skins from the peas, which is a prerequisite of the test, is a disadvantage, but the method involves the use of a small amount of readily available equipment, and untrained operators can be quickly trained to do the work. The brine flotation method is the only procedure which fractionates the sample on a maturity basis, all other methods whether chemical, physical, or mechanical give a value which indicates the average maturity of the sample. Samples could be subjected to brine separation followed by analysis by any one of these methods so as to determine the maturity make-up of the sample. A fairly good relationship was obtained between the brine flotation test and the tenderometer readings. It should be noticed, however, that Figure 6 shows greater spread for this relationship than for any of the other tests. In preparing this figure the older peas for which no values were assigned were obviously not included.

Two modifications were employed for the method of the determination of maturity by means of specific gravity, as described before. The first of these was shown previously *(11)* to give (with a shorter boiling period) good results for the determination of the maturity of lima beans. The boiling step releases gases trapped within the peas. In the earlier report on peas made by the senior author, imprisoned gas did not seem to be an important factor. Later samples, how-

ever, obtained under different conditions changed this situation.

The second of the 2 methods, in which the peas are punctured prior to the boiling step, was introduced to ensure the elimination of gases from under the skin by providing an opening for their escape. This procedure, in most cases, increased slightly the specific gravity figures obtained. This can be seen when the results of the two modifications are contrasted. The differences, however, are not great (Tables 1 and 2).

The use of a wetting agent in the water in which the peas are weighed to determine the loss in weight under these conditions, described by Lynch and Mitchell *(12)*, was not used by us, but appears to be sound in principle. It has been the experience of the workers in this laboratory, however, that adhering bubbles offer little difficulty.

The specific gravity method showed higher correlation with the tenderometer readings and the organoleptic tests than the other indices used. However, the differences between these figures and those for the correlation between tenderometer readings and organoleptic tests with alcohol-insoluble solids were not great. The specific gravity test showed the highest correlation of all the tests when the *fancy* and *extra standard* peas alone were considered, which is obviously rather important. It must be noted, however, that in the other years the values for the correlation of the specific gravity and tenderometer readings of the *fancy* and *extra standard* samples were not quite so high as those obtained in 1949, but were still slightly higher than those obtained in this range for the other methods of assessing maturity. It should be made clear that the correlation between alcohol-insoluble solids and the tenderometer readings of the *fancy* and *extra standard* samples in the other years was of the same order of magnitude as was obtained in the 1949 series.

It is desirable to mention that the limits for the specific gravities of the several grades suggested by the senior author in 1941 *(8, 9)* were later found to be too high because of irregularities which were not obvious at the time. This situation was corrected, and all other figures published from this laboratory on the applications of this method are correct. It can be stated now that these figures obtained by the boiling method should read: *fancy*, up to 1.060; *extra standard*, 1.061 to 1.075. Above this would be *standard* and *substandard*. These figures might be increased by 0.003 if the determination is based on punctured and boiled peas.

This specific gravity method lends itself well to the evaluation of the maturity of processed samples. It is rather easy of application, and requires equipment which is ordinarily available in routine and industrial laboratories.

During the course of the present work, the use of alcohol-insoluble solids was examined as a method for the measurement of the maturity of the sample. This method was originally suggested for use on canned peas *(4, 5)*. The modification *(17)* simplifies the procedure somewhat. In 1941 *(9)*, some data were presented on the use of this method for frozen peas. At that time pressure cooking of the sample of frozen peas was suggested, followed by a determination of the alcohol-insoluble solids. This method is cumbersome, but usable results can be obtained by following it. Makower, Boggs, Burr, and Olcott *(15)* presented some interesting data on the use of alcohol-insoluble solids as an index of the maturity of frozen peas. They found that this method gives satisfactory results for this purpose. This is in agreement with the results presented in this paper within the limitations of the method.

Alcohol-insoluble solids for fancy frozen peas should range up to about 13.5%, and for extra standard from 13.5 to about 16.9%.

It appears from the figures obtained in this study on total solids as an index of maturity of peas, and from other figures obtained in this laboratory in the past *(9)*, as well as from general considerations, that this method needs much further experimental testing before it can be considered for use.

The results obtained in this study for the Nielsen starch method for the assessment of the maturity of peas were based in each case on the use of starch for the standard prepared from the same variety of pea analyzed. The peas from which the starch was prepared were harvested from the same field from which the samples were harvested, and, of course, in the same year. The only variety for which this was not done was Shasta. For the 3 samples of this variety, Thomas Laxton starch was used as the standard.

The data published by McCready, Guggolz, Silveira, and Owens *(16)* indicate that the starch itself changes chemically as the peas become more mature. Also, since the composition of starch from the different varieties of peas varies, this method as an index of maturity becomes very much open to question. These variations in the starch obtained from different varieties of peas were noted in the literature, and were confirmed in this laboratory. In the examination of packaged peas obtained from the market for starch content by the Nielsen method, it would be unlikely that one would have available starches from each variety, as well as from each stage of maturity, all of which would be necessary for strictly reliable results. It is quite likely, also, that for such samples, the variety would be unknown. A further complication would present itself in the examination of samples of mixed varieties, which are sometimes encountered in practice.

Under the circumstances, it would seem hazardous to recommend this method for the determination of the maturity of peas, unless a way is found to overcome these difficulties.

The method for the determination of starch by diastase is quite cumbersome, and perhaps open to criticism —which makes it unlikely that it will be used as a method for the determination of the maturity of peas.

It can be seen from the figures presented in Table 2 that all the present methods correlate less well in the *fancy* and *extra standard* range than when the figures for all the grades of maturity are used in the calculation. This indicates the relative inaccuracies in all the methods available up to the present for the determination of the

maturity of peas, at least in the maturity ranges of greatest commercial importance. The figures in the standard error of estimate column indicate that relative inaccuracies are present, especially in the Nielsen starch method.

Makower, Boggs, Burr, and Olcott *(15)* mentioned that variation was found in the figures on specific gravity. An inspection of their results shows that similar variation can be found in the other indices listed by them, but which they did not mention specifically in this connection.

It is the present opinion of the workers in this laboratory that varietal differences in wrinkled peas have little, if any, effect on the specific gravity and alcohol-insoluble solids at the same level of maturity. An apparent segregation in the figures presented herewith may be explained by possible different speeds of the viner on the different days of harvest. This suggestion, made by Lynch and Mitchell *(13)*, is worthy of consideration.

The article by Nielsen, Campbell, Bohart, and Masure *(19)* merits examination in view of the criticisms expressed by these authors of the specific gravity method for the determination of the maturity of peas. At the time this article appeared, further work on this problem had already been started in this laboratory and it seemed advisable to complete the collection of new data before undertaking a reassessment of the methods.

These authors determined specific gravity by making use of a mixture of carbon tetrachloride and xylene as the medium. This procedure was discarded a little while after it was originally published and was replaced by water or by a very dilute salt solution *(10)*.

These authors [Nielsen, Campbell, Bohart, and Masure *(19)*] stated: "He *(Lee)* has reported a coefficient of correlation of -0.8168 ± 0.027 for the relationship between the specific gravity of defrosted peas and organoleptic quality. Examination of our data shows even poorer agreement. . . ." This statement seems to imply that the agreement is not good, but such agreement can be accepted as a useful correlation. If one plots the figures published in 1941 *(9)* the resulting graph shows reasonable agreement. It is, however, not so good as that obtained by data from either of the present modifications. These observations are not altered by the fact that the specific gravities published in 1941 should have been lower, as explained before, because relatively, the relationship are substantially the same.

The present results shown in Table 2 give a high correlation between these 2 factors, granting, however, an alteration in the method. This is further illustrated in Figure 8. Furthermore, Lynch and Mitchell *(12)*, working in Australia with canned peas found a correlation between specific gravity and alcohol-insoluble solids of + 0.95. These authors concluded that: "Experimental evidence from these investigations confirms the validity of the assessment of maturity by A. I. S. determination, but the procedure is time-consuming, and, therefore, of limited value for field and factory grading. Specific gravity and succulometer figures correlate well with maturity, but again these measures are inconvenient in practice." Nielsen, Campbell, Bohart, and Masure *(19)* gave no attention to the use of alcohol-insoluble solids as a method for the assessment of maturity of frozen peas.

The figures in Table 3 show that all the 20 replicates of Thomas Laxton peas based on either specific gravity modification confirm the tenderometer reading that the sample is in the fancy grade. Considerable variation is noted in the figures for starch by the Nielsen method, namely, from 3.00 to 3.90%. According to the standards set by Nielsen, Campbell, Bohart, and Masure *(19)* 5 of these replicates, or 25% of them, are in the *extra standard* range and the remainder are in the *fancy* range, based on total starch. The coefficient of variation using the specific gravity method (boiled) is 0.08%, the coefficient of variation using the specific gravity method (punctured and boiled) is 0.13%, and the coefficient of variation using the Nielsen starch method is 7.0%. This comparison somewhat overstates the differences in useful precision, however, in that starch changes more rapidly than specific gravity.

It seems to be hoped at the present time that an objective method will be found for peas and/or other vegetables by means of which it will be possible to place samples in definite grades with infallible accuracy. In view of the important factor of biological variation, it is perhaps unlikely that this ideal can be attained. The present methods, however, seem to be quite satisfactory for all but samples in the borderline zone. The results of the work in this laboratory and the results of that of Lynch and Mitchell *(12, 13)* indicate that the best methods available at present for the objective evaluation of the maturity of processed peas are the methods of specific gravity and of alcohol-insoluble solids. These methods together with the brine flotation test could be used to determine the maturity of borderline zone samples objectively on a majority rule basis.

SUMMARY

1. A critical study of the objective methods used in the determination of the maturity of frozen peas was made, and the results presented herewith.

2. The results show that the method of specific gravity, as modified in this paper, and that of alcohol-insoluble solids are the most reliable of the objective tests proposed to date for use on processed peas.

3. The starch method suggested by Nielsen seems impractical for general use.

4. The method of total solids requires further detailed testing before it can be considered for use.

5. It is suggested that the maturity grade of borderline zone samples be determined by using a majority rule of the three tests available at present: specific gravity, alcohol-insoluble solids, and brine flotation tests.

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Sorbic Acid as a Fungistatic Agent for Foods. VIII. Need and Efficacy in Protecting Packaged Cheese[a]

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(Manuscript received July 16, 1953)

Sorbic acid, a harmless polyunsaturated C_6 fatty acid, satisfies all the requirements of a good fungistatic agent for use on many cheese wrappers. Over 13 million pounds of cheese per year can be saved by use of suitable fungistatic wrappers. This is in addition to other benefits resulting from the use of such wrappers.

The problem of mold control is probably nowhere more omnipresent than in the cheese industry. The texture and flavor of most cheeses require that they do not dry out; hence, the cheese must be stored in a very humid atmosphere or must be enclosed in moisture-proof packages. Any air on the interior of such packages quickly reaches high humidity. With the high relative humidity and the rich substrate provided by the cheese, an ideal habitat for mold is formed. These somewhat opposing objectives of moisture retention and mold control are paramount requirements in most cheese packaging methods.

Mold causes loss of cheese and loss of quality in all phases of production, distribution, and home use. In Table 1 an attempt has been made to estimate the amount of cheese wasted annually and to point out the amount which could be directly saved by use of suitable fungistatic wrappers. These estimates incorporate suggestions of ten executives who have broad experience in the cheese industry.[b]

TABLE 1

Estimates of waste in U. S. cheese production

Type of cheese	Amount produced in U.S. in 1952[c]	Estimated % waste before consumption	Estimated % waste avoidable by fungistatic cheese packaging	Estimated amount of waste avoidable by use of fungistatic packaging
	lbs.	%	%	*lbs.*
American	870,435,000	1½ to 5	1	8,700,000
Swiss	104,535,000	3 to 5	2	2,000,000
Cream Cheese	78,045,000	½ to 2	½	400,000
Italian types	63,390,000	3 to 5	1½	900,000
Brick & Meunster	35,970,000	2 to 5	3	1,000,000
Blue Mold	11,215,000	2 to 6	1½	150,000
Limburger	6,245,000	2 to 10	0	.....
All others	25,380,000	2 to 10	1½	400,000
Total	1,195,215,000			13,550,000

[c] Amounts produced from records of National Cheese Institute as compiled from U. S. Department of Agriculture reports. (Courtesy E. Gaumnitz, Secretary, National Cheese Institute.)

This estimate of over 13 million pounds of cheese per year direct saving, possible through effective control of molds, emphasizes the immediate and very real need for such control. It is further estimated that as mold control is improved, better methods, which will

[a] The contents of this paper were presented in part at the Thirteenth Annual Meeting, Institute of Food Technologists, Boston, Massachusetts, June 24, 1953.

[b] We wish to express appreciation to the busy cheese executives who cooperated in preparing the estimates in Table 1. References can be provided if desired.

eventually reduce the overall waste by several times this amount, will result. The gain in cheese quality and in improved distribution with better mold control are difficult to measure. As illustrative of such value, it can be pointed out that mold control to reduce losses encourages longer curing periods yielding a higher percentage of high-quality aged product. Also, the maximum shelf-life of sliced process cheese which requires slicing in the the retail stores, is one to 2 weeks except in the limited cases where special means of mold control are available. In addition, it is difficult to place a dollar value on consumer dissatisfaction with a product exhibiting gross mold contamination.

In contrast to many food products, the molding or biological spoilage of cheese, once it has been made, is in the main a surface problem. Thus a fungistatic wrapping material is an effective, convenient method of preventing waste and preserving quality. A further advantage of applying a fungistatic agent by means of the wrapper is that an accurately controlled amount of fungistatic agent can be applied to give surface action without necessarily having high concentration throughout the product.

PROPERTIES

An ideal fungistatic agent for cheese wrappers should have the following properties:

a. Effectiveness.
b. Non-toxicity.
c. Mobility to contact all surfaces.
d. Freedom from taste or odor in effective amounts.
e. Ease of handling in use.
f. Stability to cheese components so that no objectionable taste, odor or color change is caused by interaction; emulsion stability should not be affected.
g. Lack of color in effective amounts.
h. Specificity of action so that normal cheese curing is permitted.
i. Resistance to oxidation, heat or light.
j. Commercial availability in pure form.
k. Suitability for detection and measurement.

To answer this need, a new fungistatic wrapper has been developed and tested on many cheese products. The combination of thermoplastic-coated cellophane[4] and sorbic acid gives mold control in addition to moistureproofness, mechanical protection, cleanliness, ease of sealing, gas impermeability, and display value. Packaging material of this type has proved practical for over a decade of use in cheese wrapping. It was particularly chosen for this development because the heavy coating provides a vehicle for the fungistatic agent and also assures good heat-sealing of the packages.

Sorbic acid was chosen from among the many materials tested because of the following properties:

a. It was found to afford effective inhibition of mold growth by use of 2.5 g. of sorbic acid per 1000 sq. in. of wrapper *(11)* for small packages. When added directly to melted process cheese, 0.005% to 0.01% retards but does not prevent mold growth, while 0.05% was fully effective *(11)*.
b. Sorbic acid has been shown to be harmless as a component of foods *(2, 3)*, a particularly outstanding quality in comparison to other fungistatic agents. In fact, experimental results indicate that sorbic acid is actually an assimilable nutrient *(3)*, the animal organism utilizing this polyunsaturated C_6 fatty acid in the same manner as the normally-occurring saturated C_6 fatty acid, caproic acid. On the basis of 2.5 g. of sorbic acid per 1000 sq. in. of wrapper and assuming complete migration *(7)* of the fatty acid into the cheese product, a safety factor of more than 450 times would obtain. The highest ratio of wrapper to cheese exists in the popular commercial package (3¼ x 3½ x 1¼ inches) used in packaging one-half pound of cheese. The cheese at the most would contain 0.042% of sorbic acid or 94.4 mg. per half pound. If one were to eat 5½ ounces of such cheese each day, 21% of the total daily intake of 3000 calories, the individual would ingest 66 mg. of sorbic acid. Assuming 600 g. of solids in the human daily ration, this ingestion of sorbic acid would amount to 0.011% on a total solids basis. In the rat and dog, sorbic acid as 5% of the solids in the ration was harmless *(2)*. The safety factor would be more than $\frac{5.000}{0.011}$ or 450 times. When the highest level of sorbic acid on the wrapper is employed for special cases, 5.0 g. per 1000 sq. in., the safety factor is still more than 200 times. The data on harmlessness of sorbic acid *(2, 3)* have been accepted by U. S. Food and Drug Administration as proof of non-toxicity *(12)*.
c. The mobility or diffusion rate of sorbic acid in cheeses is relatively low, and its water solubility is only about 0.2% at 20° C. *(1)*. This low water solubility is advantageous, however, because it permits a high surface concentration when the wrapper is placed in contact with the cheese and limits diffusion so that an effective surface concentration is maintained for sufficient time to protect the cheese *(11)*.
d. The minimum taste perception in mild process cheese was established as being approximately from 0.2 to 0.5% concentration, or about 4 to 10 times that of an effective fungistatic concentration *(11)*. Flavor tests of mild and sharp American, Swiss, brick, Italian Provalone and blue cheese in contact with wrappers carrying for the most part 5 g. of sorbic acid per 1000 sq. in. showed no flavor differences. Differentiation between cheese slices adjacent to control and sorbic acid-treated wrappers, the latter treated with the highest concentration of 5 g. of sorbic acid, was not statistically significant *(11)*. The odor of the cheeses was not changed by addition of sorbic acid in concentrations which would result from wrapping cheese in treated wrappers *(11)*. The pH change upon addition of fungistatic amounts of sorbic acid to cheese is too slight to affect taste *(11)*.
e. Sorbic acid is an easily handled light colored crystalline material. It does not create unusual difficulties in application to the wrappers, nor in the use of such treated wrappers.
f. Sorbic acid does not change the color, texture, odor or taste of cheese stored in sorbic acid-treated wrappers and held for extended periods at different temperatures *(11)*. Melnick and Luckmann *(7)* have obtained nearly 100% recovery of sorbic acid from wrapped cheese under favorable conditions. Under unfavorable conditions, sorbic acid is metabolized by molds to carbon dioxide and water by the same enzyme mechanisms operating in the animal organism *(9)*.
g. Sorbic acid does not change the color of the products wrapped.
h. Sorbic acid exhibits a selective action against certain bacteria and yeasts, which fortunately does not appear to interfere with normal cheese curing in treated wrappers. Phillips and Mundt *(10)* reported inhibition of yeast scums and mold by sorbic acid but no effect on bacterial curing of dill pickles. Emard and Vaughn *(4)* have reported studies on the selective action of sorbic acid-containing media on bacteria. The α, β-unsaturated fatty acids, such as sorbic acid, act as fungistatic agents in that as normal transitory metabolites in the oxidation of saturated fatty acids by molds, a high initial concentration is capable of inhibiting the dehydrogenase enzyme system in molds *(9)*.
i. Sorbic acid is sufficiently resistant to oxidation and sublimation that it and the treated wrappers may be stockpiled for extended periods *(8)*. In wrapped cheese stored

[4] Developed in this laboratory.

for 6 weeks at 45° F., oxidation of the sorbic acid applied to the surface does not occur. Sorbic acid is no more susceptible to oxidation than the polyunsaturated fatty acids in butter fat *(8)*.

j. Pure sorbic acid is now commercially available for use in cheese wrappers *(5, 11)*.

k. An accurate ultra-violet spectrophotometric method for detection and measurement of sorbic acid in cheese has been developed and tested on the main cheese types *(6)*.

TECHNOLOGICAL EVALUATION

In this point-by-point examination, it is evident that sorbic acid is well suited for use in protecting cheese products from undesirable mold growth. Tests of sorbic acid-bearing wrappers on commercial wrapping equipment show that it is practical to use and gives effective mold inhibition when compared with control samples without the fungistatic agent.

It has also been shown *(11)* that proper application of coated cellophane carrying sorbic acid is actually fungicidal when some types of process cheese are packaged. Under these conditions, cheese so packaged will keep for at least 8 months without molding. However, if during the latter part of this period, the package is broken and the cheese reinoculated, the new inoculant will grow. This demonstrates diffusion and gradual loss of sorbic acid in the packaged product. This lack of permanent fungicidal action is a desirable protection to ultimate consumers in case of breakage or mishandling of packaged goods since subsequent mold growth will give visible warning of possible spoilage.

From the foregoing enumeration of properties it is evident that sorbic acid-treated wrappers must be properly used; the following conditions of packaging have been found essential for successful mold inhibition:

a. The wrapper must be sealed to prevent recontamination after incumbent mold is inhibited or destroyed.

b. The wrapper and the cheese must have close contact so that an effective concentration of sorbic acid on the surface of the cheese can be set up quickly and maintained for an effective time.

c. The initial mold contamination of the cheese must be relatively light (no relaxation of good sanitary practices) in order to obtain the high sorbic acid-mold dehydrogenase enzyme ratio which is evidently necessary for the inhibitory action *(9)*.

Samples of sliced process cheese packaged on commercial vacuum equipment showed 35% failure in the case of the controls and 1% in the case of the sorbic acid-treated samples after 8 months at 32° F. Other commercially hand-wrapped samples showed 60% moldy on controls and none on those with sorbic acid-treated wrappers* after 2 months at 45° F. *(11)*. One previous run of this hand-wrapping test was a failure because of poor closures of the packages *(11)*. Tests of packaging natural American cheese indicate that more sorbic acid is required than for sliced process cheese, evidently because of the greater enzyme activity in the natural (unheated) cheese, but that 4 g. per 1000 sq. in. of wrapper is effective. Lower amounts of sorbic acid give retardation but not complete inhibition of molding *(11)*.

* Actually one sample became moldy but this was due to breakage of the seal.

SUMMARY

Tabulated estimates show that over 13 million pounds of cheese per year can be saved directly by suitable fungistatic wrappers. Quality and distribution gains are also apparent. Eleven requirements of a good fungistatic agent for use on cheese wrappers are presented. Sorbic acid is a new, effective, non-toxic and practical fungistatic agent specifically adapted for cheese packaging; it satisfies all the requirements listed. It has been shown to be very useful when carried on the inner surface of thermoplastic-coated cellophane cheese wrappers. Suggestions for effective use of these wrappers emphasize desirability of good closures, maximum wrapper-to-cheese contact and avoidance of high initial mold contamination. Careful tests of sorbic acid-treated wrappers on laboratory and commercial scale production have demonstrated that the fungistatic wrapper will increase the shelf-life and result in a much lower percentage of spoiled packages.

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Pasteurization and Storage of Sweetened and Unsweetened Lime Juice[a]

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The effects of different heat treatments were judged on the basis of bacterial counts, flavor, destruction of pectinesterase enzyme, and cloud stability. A heat treatment of 150° F. (65.6° C.) was effective in reducing the number of microorganisms to a low level, but 170° F. (76.7° C.) was required to destroy pectinesterase and insure cloud stability in 35° F. (1.7° C.) storage. Under refrigeration, flavor of the heated products remained practically unchanged for 15 months, but some changes were observed in the unheated samples. At 80° F. (26.7° C.) all samples deteriorated rapidly.

Canning of lime juice has never achieved commercial significance mainly because the products deteriorated rapidly at room temperature. The methods of preparation used have been those developed for orange and grapefruit juices *(2, 4, 5, 7, 12, 16, 17, 19)* and it was thought that some variation in the pasteurization procedure might improve quality. Lime juice is much more acid than orange or grapefruit juices, and it would be expected that a somewhat lower temperature of pasteurization would suffice. Typical total acidity values for Persian (Tahiti) limes range from 5 to 7% calculated as citric acid, while Florida oranges range from 0.7 to 1.2% and grapefruit from 1.0 to 1.6%.

Refrigerated storage at 35° F. (1.7° C.) also seemed to offer possibilities as a means of preserving the lime products until used, since room temperature storage had not been successful.

Kulkarni *(9)* in 1930 discussed the preparation of clarified, sterilized lime juice. Aiyar *(1)* in 1937 prepared both sweetened and unsweetened clarified lime juices using sodium salicylate as the preservative. Kassab *(8)* studied the effect of heat as an aid to clarification by filtration and concluded that heating to 160° F. (71.1° C.) and cooling prior to filtration resulted in the least sediment in storage. Pruthi and Lal *(15)* have investigated the retention of vitamin C in pasteurized and chemically preserved lime juice. They observed that deaeration combined with flash pasteurization at 195 to 205° F. (90.6 to 96.1° C.) gave the best results.

EXPERIMENTAL METHODS

Extraction of lime juice and preparation of experimental samples. Persian limes obtained from groves in the Ridge District of Florida were processed in the laboratory pilot plant. Fruit was immersed in water and washed on a rotating brush washer. Juice was extracted with a Rotary Juice Press[c] and excess pulp and seeds removed by a cylindrical screw-type finisher equipped with a screen having 0.040 in. perforations. The juice (approx. 50 gal.) was cooled as extracted to 40° F. (4.4° C.) in a cold-wall tank and maintained at this temperature until heat processed. Contents of the tank were thoroughly mixed and divided into 2 portions. One portion was processed and canned unsweetened; sugar was added to the other portion to a soluble solids content of 45° Brix. This sweetened product when diluted with 4½ parts of cold water gave an excellent limeade, ready for drinking.

Both the unsweetened and sweetened products were subjected to various heat treatments and canned. Portions of each were heated to temperatures of 120°, 130°, 140°, 150°, 160°, 170°, 180°, 190°, and 200° F. (48.9°, 54.4°, 60.0°, 65.6°, 71.1°, 76.7°, 82.2°, 87.8°, and 93.3° C.) respectively, by pumping the juice through a stainless steel tubular, water heated pasteurizer. The pasteurizer tube was 33 feet long, 5/16 in. outside diameter and 0.24 in. inside diameter. It was coiled in a chamber through which hot water was rapidly circulated at controlled temperature.

The juice was heated in 17 sec., held at the specified temperature for 5.7 sec., filled into 4-ounce cans in 7.5 sec., closed in 7.5 sec., held inverted for 15 sec., and then cooled by rotating the cans under cold water sprays. Samples were stored at 35 and 80° F. (1.7 and 26.7° C.) and examined periodically for loss in cloud and flavor. Some samples were stored at 0° F. (−17.8° C.) for future reference. Initial observations of bacteriological plate counts, pectinesterase enzymes, and cloud density were made.

Freshly extracted juice used in preparing the experimental packs had a total acidity of 5.75% citric acid, a pH value of 2.38, soluble solids content of 8.8° Brix, recoverable oil of 0.032%, and suspended solids of 8% by volume.

Methods of analysis. Initial microbial populations in the lime juice samples were determined by preparing suitable dilutions and plating 1 ml. portions in duplicate on Lindegren's agar *(14)* and acid-sucrose agar *(13)*. The acidities of the media were pH 5.8 and pH 3.8 respectively. Plates were observed after 48 to 72 hours incubation at 37° C.

Pectinesterase activity (PE.u.) was determined by the method of MacDonnell, Jansen, and Lineweaver *(11)*, with a modification suggested by Jansen *(6)*. The method consisted essentially in titrating a 2- to 6-ml. sample, depending on its relative enzyme activity, with 0.1N sodium hydroxide to pH 7.5 in the presence of a 20-ml. portion of 1% pectin solution in 0.25 molar sodium chloride. Sufficient water was added prior to pH adjustment so that on addition of the required amount of sodium hydroxide solution, the total volume was about 40 ml. From time to time as the enzymatic liberation of acid took place, 0.02N sodium hydroxide was added to readjust the pH to 7.5. From the amount of standard alkali used, the milliequivalents of ester hydrolyzed were calculated. The results are reported as (PE.u.) ml. x 10^5. The symbol (PE.u.) ml. represents milliequivalents of the ester hydrolyzed per minute per ml. sample.

Observations for cloud index were made by the method of Loeffler *(10)* in which the samples were centrifuged for 10 minutes and the light transmission determined with an Evelyn[c]

[a] Presented at the Thirteenth Annual Meeting of the IFT, Boston, Mass., June 23, 1953.

[b] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

[c] The mention of equipment and products does not imply that they are endorsed or recommended by the U. S. Department of Agriculture over other equipment or products having the same or similar properties.

colorimeter, using a No. 720 filter. The values have been expressed as cloud density (2-log % light transmission). Decreasing cloud density values indicate loss of cloud or suspended matter.

All data are presented as taken for both sweetened and unsweetened juices; in making comparisons it should be noted that the addition of sugar resulted in an increase in volume of 45%.

RESULTS AND DISCUSSION

Microbiological plate counts on Lindegren's agar are given in Table 1. The initial count of 2000 in the juice is quite low in comparison with the counts generally found in other citrus juices. Heat treatment as low as 120° F. (48.9° C.) produced a marked reduction in the numbers of microorganisms. Further reductions were found with increasing temperatures until, at 150° F. (65.6° C.) and above, plate counts showed that most if not all were destroyed. Acid-sucrose agar was used to favor the growth of organisms, especially yeasts, capable of growing in high-sugar high-acid media. Counts obtained with this agar are not given in the table as they follow the same trends as found with Lindegren's agar. Counts on acid-sucrose agar averaged about half those on Lindegren's agar. Most plate counts were lower with sweetened than unsweetened juices, but more data would be needed before significance could be attached to these differences.

TABLE 1

Effects of heat treatments on microbial counts and pectinesterase activity in lime juices

Heat treatment	Unsweetened juice			Sweetened juice		
	Plate count Lindegren's agar	(PE.u.) ml. x 10^4	% Enzyme inactivation	Plate count Lindegren's agar	(PE.u.) ml. x 10^4	% Enzyme inactivation
Unheated	2000	15.0	00	500 [a]	9.7	00
120° F.	390	15.0	00	160	9.0	7
130° F.	130	14.0	7	20	7.6	21
140° F.	50	10.3	31	10	6.6	32
150° F.	0	2.1	86	0	5.0	49
160° F.	0	0.0	100	5 [a]	1.5	84
170° F.	0	0.1	99	0	0.5	95
180° F.	5 [a]	0.3	98	5 [a]	0.3	96
190° F.	5 [a]	0.3	98	0	0.3	96
200° F.	5 [a]	0.3	98	5 [a]	0.4	96

[a] Too few on agar plate for accurate count.

Samples were placed in storage at 35 and 80° F. (1.7 and 26.7° C.) to observe for the development of fermentation. In no case did any swells develop. Too much reliance should not be placed on this particular result as previous experience has shown that unheated lime juice may or may not ferment.

Pectinesterase values are given in Table 1 as units of activity and as percentage inactivation. Heat treatments of 120° F. (48.9° C.) had no significant effect on enzyme activity and heating to 130° F. (54.4° C.) had only a slight effect; but with increasing temperatures in the range of 140 to 160° F. (60.0 to 71.1° C.) greater percentages of the enzyme were destroyed. At 170° F. (76.7° C.) and above, the residual pectinesterase was too small to be of significance. The differences at low activity levels are within the accuracy of the method. One of the principal limitations is in measuring minor shifts in pH over a period of a half hour or more. In this study average error between duplicates was 2.2% of the original enzyme value. Some differences will be noted between the values obtained with the unsweetened and sweetened products, but no special significance is attached to these differences. The enzyme values in the freshly extracted juice are of the same order as those found in orange and grapefruit juices in this laboratory *(18)*.

Effects of heat treatments on cloud density of undiluted lime juice products stored at 35° F. (1.7° C.) are shown in Table 2. Low figures indicate comparatively high transmissions of light and very little remaining cloud. An adequate amount of well dispersed finely divided particles or cloud is generally associated with good consumer acceptance in citrus juices. The characteristics of citrus juices are closely related to cloud, as completely clarified juices are markedly lacking in distinctive flavor. It had been hoped that examination of diluted juices would give a direct comparison between sweetened and unsweetened samples, but light transmissions were so high that differences were beyond the sensitivity of the method. It was therefore apparent that observations should be limited to undiluted samples. Control values were obtained by using samples of the various packs that had been stored at 0° F. (−17.8° C.) for a month.

TABLE 2

Cloud density values on undiluted lime juices stored at 35° F.

Heat treatment	Cloud density values [a]					
	Unsweetened juice			Sweetened juice		
	Control [b]	2 mos.	15 mos.	Control [b]	2 mos.	15 mos.
Unheated	0.70	0.48	0.28	0.31	0.30	0.28
120° F.	0.68	0.52	0.21	0.35	0.31	0.38
130° F.	0.66	0.52	0.12	0.36	0.26	0.31
140° F.	0.68	0.55	0.28	0.36	0.31	0.32
150° F.	0.75	0.68	0.80	0.35	0.27	0.32
160° F.	0.77	0.75	0.82	0.36	0.30	0.30
170° F.	0.75	0.80	0.82	0.36	0.36	0.32
180° F.	0.75	0.77	0.82	0.37	0.36	0.36
190° F.	0.82	0.77	0.82	0.35	0.34	0.33
200° F.	0.82	0.80	0.82	0.36	0.34	0.33

[a] Cloud density = 2 — log (% light transmission).

[b] Control values indicated were taken on materials stored one month at 0° F.

Satisfactory cloud retention was observed in the unsweetened samples treated at 160° F. (71.1° C.), the heat treatment required to destroy 95% or more of the enzyme activity. Samples sweetened to 45° Brix by the addition of sucrose were observed to clarify immediately on solution of the added sugar thus giving low cloud density values as shown in Table 2. The effect of sugar in clarifying the lime juices overshadowed any effects of the enzyme on cloud during storage.

In order to be sure this effect was not solely one of dilution, 19 samples of unsweetened lime juice were diluted with water to the same volume they would occupy if sweetened and cloud densities determined. The average cloud density before dilution was 0.82 and after dilution 0.64, a decrease of 0.18. The samples involved in this study showed an average cloud density of 0.74 without sweetening and 0.36 with sweetening.

a decrease of 0.38. This indicates that the sugar did have a clarifying effect other than by dilution.

Unheated lime juice did not clarify as rapidly as unheated orange and grapefruit juices which normally show 90% or more clarification within a week at 35° F. (1.7° C.) *(3)*.

Examination of lime juices stored at 80° F. (26.7° C.) for 4½ months indicated that they had undergone marked changes. The unsweetened samples that had been heated to 160° F. (71.1° C.) or less had completely clarified and a fibrous coagulum had formed which was difficult to disperse. Samples that had been heated to 170° F. (76.7° C.) or above showed a milky appearance not characteristic of the fresh juice and a loose coagulum had formed which could be readily dispersed.

The sweetened samples had all become clear when prepared; however, during storage at 80° F. (26.7° C.) for 4½ months some material settled out in products heated to 150° F. (65.6° C.) or lower with only a trace of sediment in those heated to 160° F. (71.1° C.) or higher temperatures. All samples had developed a strong terpeney or "medicinal" flavor during this storage period at 80° F. (26.7° C.) and were considered unacceptable. The storage life of these products at room temperature would be limited.

At 35° F. (1.7° C.) good flavor retention was observed in all samples. No flavor change was noted in heat treated samples stored for 15 months at this temperature. There was some change in the flavor of the unheated products. Heat treatments used in this study did not appreciably injure the flavor of the lime juice products.

Currently, pasteurization temperatures of 180 to 205° F. (82.2 to 96.1° C.) are in general use in commercial canning of orange and grapefruit juices, the actual temperature depending upon the type of the equipment, the acidity of the fruit, and its condition. These studies indicate the possibility of attaining satisfactory stability of lime juice with somewhat lower pasteurization temperatures, but no injurious effects were noted from the use of the higher temperatures. It is apparent that sufficient heat treatment to inactivate pectinesterase enzymes and destroy bacteria is desirable and that refrigerated storage favors retention of highest quality in the product.

SUMMARY

Unsweetened lime juice, 8.8° Brix, and a portion of the same juice sweetened to 45° Brix have been heated to temperatures ranging from 120 to 200° F. (48.9 to 93.3° C.) canned, cooled, and tested for stability in storage at 35° F. (1.7° C.) and 80° F. (26.7° C.).

Most if not all of the microorganisms contained in the juice were destroyed by heat treatments of 150° F. (65.6° C.) and higher.

Heat treatments of 160° F. (71.1° C.) or above reduced the pectinesterase activity of unsweetened juice to 5% or less of that initially present and gave satisfactory cloud retention in 35° F. (1.7° C.) storage. Much of the cloud disappeared when sugar was added in preparing the sweetened samples and this overshadowed any clarifying effects of pectinesterase during storage.

Severe off-flavor development was observed in all samples at 80° F. (26.7° C.) in 4½ months and marked changes in appearance were observed in the unsweetened samples.

Good flavor stability was obtained at 35° F. (1.7° C.) for periods up to 15 months with no noticeable change in the flavor of the heated samples and with small changes in the unheated.

These results indicated that pasteurization temperatures used in this study did not appreciably injure the flavor of the lime juice products but were of benefit in stabilizing the cloud, particularly in the unsweetened samples. Refrigerated storage was essential for maintenance of quality for both sweetened and unsweetened products even for the brief time of 4½ months.

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A Micro-Method and Apparatus for the Multiple Determination of Rates of Destruction of Bacteria and Bacterial Spores Subjected to Heat[a,b]

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A micro-method and an apparatus for determining the thermal resistance of bacteria and bacterial spores are described. The apparatus treats ten or more samples simultaneously and can be used over a wide range of temperatures. The bacteriological procedure utilizes the apparatus described. Data can be accumulated rapidly and in sufficient quantity to establish confidence limits of the slopes of rate-of-destruction curves.

The first systematic study of the thermal resistance of bacteria and bacterial spores to temperatures greater than 100° C. (212° F.) was that of Bigelow and Esty *(1)*, who introduced the procedure of heating bacterial suspensions in sealed glass tubes immersed in an oil bath. Since 1920 many modifications of their method, as well as several new and different methods for the determination of the thermal resistances of microorganisms, have been reported in the literature.

At present five methods are in general use for the determination of thermal death-time curves of bacteria and bacterial spores. These methods may be classified as the thermal death-time tube method *(1)*, the thermal death-time can method *(6, 15)*, the rate of destruction method *(17)*, the thermoresistometer method *(5, 11)*, and the miniature retort method *(9)*.

Tanner *(14)* has reviewed most of the early literature on the subject and Stumbo *(12)*, in a comprehensive review of the literature in the field, has discussed in detail the procedures and limitations of most of the various methods.

With all the methods except that which measures the rate of destruction *(17)*, the data obtained represent the end-point of destruction. These data may be analyzed by methods presented by Stumbo *et al. (13)*, Pflug and Esselen *(5)*, and Schmidt—as described by Reynolds *et al. (7)*—to obtain D[c] values for the organisms studied.

However, Viljoen *(16)* and Reynolds and Lichtenstein *(8)* were able to obtain data regarding rate of destruction by using interval counts on thermal death-time tubes. Some error may have been introduced by the failure to culture the entire contents of the thermal death-time tubes. Furthermore, the thermal death-time tube and can methods have recently been considered " . . . inapplicable for the determination of accurate thermal death times if conditions are such as to require significant corrections to be made for temperature lags" *(12)*.

The apparatus described by Williams *et al. (17)* for the determination of spore-destruction rates is limited in its application, as it cannot be used at temperatures much above 121.1° C. (250° F.), because of the time necessary to bring the relatively large volume of suspension used in this method to the desired temperature.

In 1952, Stern *et al. (10)* presented data demonstrating that a small volume of suspension (0.01 ml.) located in a thin-walled glass capillary (0.8 mm. inner diameter) would have the heat penetration characteristics of an infinite cylinder. The system, if utilized for thermal death-time studies, would have the advantage of short heating and cooling lag times. In addition, the heat penetration characteristics at locations other than the central axis could be calculated by use of the charts of Gurney and Lurie *(3)*, and the heat penetration characteristics on the axis could be determined by use of the apparatus described by Stern *et al.*, or by use of the tables presented by Olson and Schultz *(4)*. Therefore, lag factors and correction times could be accurately determined, if desired.

An apparatus has been developed in the Department of Food Technology at the Massachusetts Institute of Technology that utilizes the system previously described by Stern *et al. (10)* to obtain data on rates of destruction of bacteria and bacterial spores over a wide range of temperatures (from below 100° up to 140° C.—212° to 284° F.). The apparatus is relatively simple in construction and operation, and comparatively inexpensive to construct. It can be built at a cost of approximately $100, exclusive of the temperature-controlling devices. Ten or more samples can be treated in the apparatus simultaneously, and all samples subjected to a particular bath temperature receive identical heating and cooling treatments.

Sufficient data can be obtained by use of the apparatus to establish statistical confidence limits of the slopes of the curves representing rate of destruction. The samples can also be treated in such a manner as to obtain data on end point of destruction, if desired.

DESCRIPTION AND OPERATION OF APPARATUS

A detailed diagram of the apparatus is shown in Figure 1. Two 2-quart, stainless steel sauce pans, 7⅝ inches in diameter and 4 inches in height, are used to contain the heating and cooling baths. The pan for the hot bath contains an electric mixer, a thermostatic controlling element, one or more thermometers, and a heating element. The pans are held rigidly in a brass framework in such a manner that the distance between the centers of the baths is 18¼ inches. The brass bands around the

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[c] D is a representation of the slope of a rate-of-destruction curve. It is the time at a particular temperature required to accomplish a 90% reduction in number of spores *(13)*.

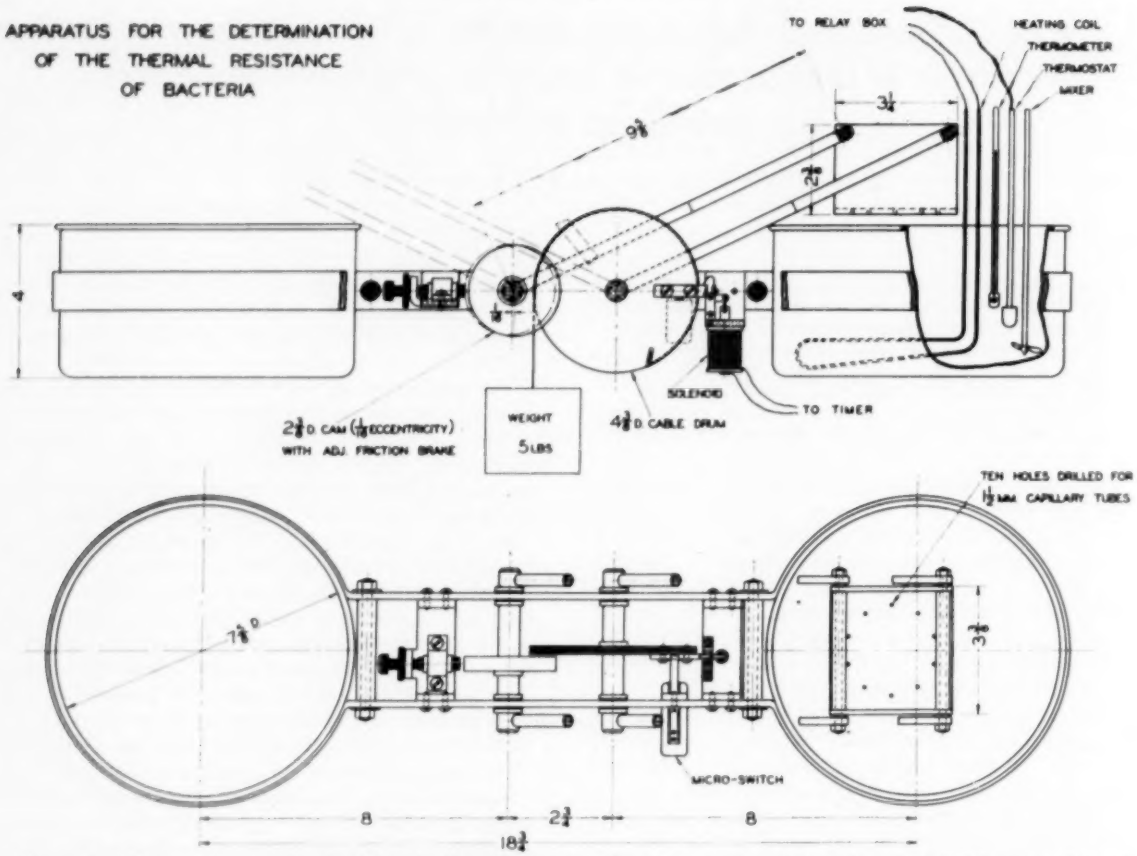

Figure 1. Diagram of apparatus for determination of accurate rates of destruction of bacteria and bacterial spores when subjected to heat.

pans are kept from spreading by *2* bolts in the framework, located near the pans.

A tray of ⅛-inch aluminum, 3⅛ inches in width and 3¼ inches in length, through which 10 or more holes are drilled, serves as a carrier of samples. The carrier has *2* sides of ⅛-inch aluminum, 3¼ inches in length and 2¾ inches in height.

Aluminum rods, each 9⅝ inches long with milled ends, are pivoted ¼ inch from the edge of each of the upper 4 corners of the sides of the sample carriers. The lower ends of the forward set of aluminum rods are pinned near the ends of a brass dowel, which passes through but is free to rotate in the brass framework. The center of this dowel is 8 inches from the center of the hot bath. The rear set of aluminum rods are attached near the ends of a second brass dowel, the center of which is 2¾ inches from the center of the first brass rod. This arrangement results in parallel motion of the aluminum rods and insures that the carrier tray will always be horizontal in traversing the semicircular path from bath to bath. As a result, the sample tubes are always in a vertical position, and centrifugal force (which would tend to throw the tubes outward, thereby causing breakage) is minimized.

The driving mechanism is a cable drum, 4⅛ inches in diameter, which is attached close to the center of the brass dowel nearest the hot bath. A 5-lb. lead weight is attached to the end of a cable affixed to the cable drum. A small piece of aluminum with a bevelled edge is affixed to the side of the drum.

A solenoid assembly is located between the cable drum and the framework bolt. The solenoid is a standard, rapid-acting, 110-120 volt A. C., hollow-cored solenoid. The core of the solenoid is modified with a hinged catch, arranged as shown in Figure 1. A lever arm micro-switch is affixed to the brass framework in such a manner that the arm is depressed by the bar of the cable drum when the sample carrier is in the hot bath.

The braking mechanism consists of a brass eccentric cam, 2⅛ inches in diameter, with 1/16-inch eccentricity, and an adjustable friction brake. The eccentric is pinned to the brass dowel nearest the cold bath. The brake is a Micarta cylinder, and its position with respect to the eccentric is controlled by the adjustment of a large screw. The action of the eccentric and the cylinder is similar to that of a wedge and is adjusted so as to stop the carrier assembly just before the aluminum arms strike the edge of the cold bath. In proper adjustment, the distance between the arms and the edge should be 1/16 inch.

The operation of the apparatus can be best understood from the description of the procedure used for a typical run. First, the timer is set at the desired time of exposure, and the tubes (which will be discussed later) are placed in the holes in the carrier tray, which is held in position over the cold bath. A cover of thin sheet aluminum is snapped over the carrier tray to prevent the tubes from riding up in the holes upon entrance into the baths. The carrier assembly is then manually pushed toward the hot bath, and the tubes automatically enter this bath. During this operation the cable drum rotates, raising the lead weight. As this occurs, the aluminum bar on the side of the drum depresses the microswitch starting the electric timer. At the same time the bar is caught under the hinged latch affixed to the core of the 110-120 volt solenoid, thus preventing the weight from falling. The eccentric cam is also rotated during this operation, so that space is present between the eccentric and the friction brake.

A schematic diagram of the electric timing device is presented in Figure *2*. A difference in potential of 3 volts is maintained between the hands of the clock and the setting hand. When contact is made between the clock hand and the setting hand, current flows through a small solenoid. The solenoid action

closes a switch and allows the line current to flow through the hollow-cored solenoid.

The activation of the 110-120 volt solenoid causes the core to be drawn into the body of the solenoid. This, in turn, pulls the hinged latch away from the aluminum bar and allows the drum to rotate as the 5-lb. lead weight falls free. The rotation of the cable drum drives the carrier assembly out of the hot bath and into the cooling bath from 0.37 to 0.40 second (as determined by stroboscopic analysis). The eccentric is also caused to rotate by the movement of the carrier assembly. As the cam rotates, it contacts the Micarta cylinder. The wedging action results in a comparatively gentle stopping of the carrier assembly before the aluminum arms contact the edge of the cooling bath.

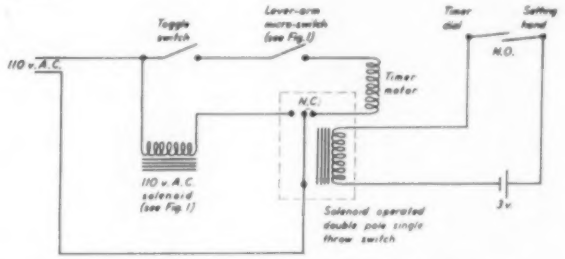

Figure 2. Schematic diagram of electric timing device.

EXPERIMENTAL

The tubes used as sample containers are standard blood coagulation tubes (capillary), with an inner diameter of 0.8 mm., an outer diameter of 1.5 mm., and a length of 3 inches. The tubes are soaked for 3 hours in a potassium dichromate—concentrated sulphuric acid solution, rinsed thoroughly in cold running tap water, then rinsed in distilled water, and finally dried in a hot air oven. One end of each tube is sealed and bent in a gas flame from a Bunsen burner with a wing tip.

The tubes, in groups of 25, are then packed in 15 x 125 Pyrex test tubes, which are covered with aluminum foil, and sterilized overnight in a hot air oven at 170° C. (338° F.). After the tubes are removed from the oven and cooled, 0.01 ml. of a bacterial suspension is added through the open end of each tube. For this purpose, a tuberculin syringe graduated in hundredths of a millimeter and a 24-gauge, 1½-inch needle are used. The open end of each tube is then sealed in a gas flame, either by forming a bead or by drawing at the tip of the tube. The cylinder of solution that is formed is approximately ½ inch long and is located, after the sealing of the tube, about ½ inch from the drawn or beaded end of the tube.

All the tubes for a day's run are prepared at once. Between 100 and 150 tubes can be filled and sealed by one person in 2½ hours.

Ten tubes taken at random (to randomize volumetric errors) from all the tubes that have been prepared, are placed in the carrier tray of the apparatus with the bent ends of the tubes uppermost. The bent ends prevent the tubes from falling through the holes in the carrier tray. The aluminum cover is snapped over the carrier tray, and the tubes are subjected to the specific temperature-time relationship desired for the run.

Between runs, the filled and sealed tubes that are not in use are stored in a refrigerator. When removed from the refrigerator for use in an experimental run, the tubes are allowed to warm to room temperature before being placed in the carrier tray of the apparatus.

After the tubes are heated and cooled, they are removed individually from the carrier tray and wiped with a clean, sterile, cellulose wipe. Each tube is then placed in acetone for 5 minutes (to remove any of the heating medium that may be adhering to the surface) and then in a potassium dichromate—concentrated sulphuric acid solution for 6 minutes.

Next, each tube is held under cold running tap water for 3 minutes and finally rinsed with 50 ml. of sterile distilled water. Following this treatment, each tube is immediately placed in an individual, sterile, 16 x 150 Pyrex culture tube (with a cotton plug) to which 5.0 ml. of sterile distilled water is added by means of a sterile volumetric pipette. Throughout the cleaning operation, the capillary tubes are handled only by sterile forceps.

Each capillary tube is then broken and crushed in its culture tube by means of a sterile glass rod with a flattened end. After the capillary tube is broken, the glass rod is moved in a spiral motion 25 times, which pulverizes the glass from the capillary tube. The rod is then moved up and down in the water 100 times to disperse the microorganisms uniformly throughout the liquid.

Aliquots are then plated according to standard bacteriological techniques for diluting, plating, and counting. Triplicate plates containing a 1.0-ml. aliquot each of an appropriate dilution are poured for all tubes where the survivors are estimated to be greater than 10%, duplicate plates containing a 2-ml. aliquot each where the survivors are estimated to be between 10 and 5%, and one plate containing a 4-ml. aliquot where the survivors are estimated to be 5% or less.

Ten control capillary tubes are used for each temperature run during a day. The control tubes are chosen at random from all the sample tubes prepared on the day, placed in the carrier tray in position over the cold bath, and subjected to the same cleaning, crushing, and plating procedures as those used for the sample tubes. One control tube is plated with each set of 10 sample tubes. Sterile, empty, sealed capillary tubes have been carried through the same procedures used for the control tubes and have never shown contamination either by the organism under study (a thermophilic flat sour organism) or by other organisms that would grow under the conditions of media and incubation in a study of a thermophilic flat-sour organism.

No problem of sampling is involved in the estimation of the number of organisms, because all the suspension subjected to a particular treatment is used in the preparation of the dilutions for plating.

Typical experimental run. In a typical experimental run the test organism was *Bacillus stearothermophilus* No. 1518. The original culture was obtained from the Research Laboratories of the American Can Company. The culture was grown on nutrient agar slants for 7 days at 55° C. (131° F.). A portion of 1 ml. of sterile distilled water was added to each slant, and the surface of each slant was gently scraped with a sterile inoculating loop. The liquids from the slants were pooled and filtered through sterile glass wool into a sterile, 4-oz., screw-cap bottle containing several sterile glass beads. The filter was washed with 15 ml. of sterile distilled water, and the washings were combined with the filtrate. The suspension was stored in a refrigerator until needed.

For the experimental run, 1 ml. of the spore suspension was added (after vigorous shaking of the screw-cap bottle) to a sterile, cotton-plugged, 13 x 100 Pyrex test tube, which contained 1 ml. of M/15 neutral phosphate buffer (pH 7.0). The bacterial suspension in buffer was then heated for 20 minutes at 100° C. (212° F.) by suspending the test tube in boiling water. At the end of this period the tube was removed from the boiling water bath and immediately cooled by being held under cold running tap water.

Petroleum jelly was used as the heating medium and was maintained at a temperature of 131.5° C. (268.7° F.), controlled to ± 0.5° C. by a bimetallic thermostatic element. The temperature of the water in the cold bath was maintained at 26° C. (79° F.). The accuracy of the electric timer was ± 0.0025 minute.

The plating medium was Difco Dextrose Tryptone Agar. The plates were incubated at 55° C. (131° F.) for 48 hours before being counted.

RESULTS AND DISCUSSION

Data obtained in the typical instance in which the test organism *Bacillus stearothermophilus* No. 1518 was subjected to 131.5° C. (268.7° F.) are presented in Table 1. The data are expressed as the mean number and the mean percentage of surviving organisms for each series of ten tubes.

The large number of experimental values obtained permitted the data to be subjected to regression analysis.

TABLE 1
Spore destruction rates of Bacillus stearothermophilus No. 1518 in M/15 phosphate buffer (pH 7.0) at 131.5° C. (268.7° F.)

Controls
Series A—2600/0.01 ml.
Series B—2900/0.01 ml.

Time in bath, sec.	Series	Surviving organisms [a]	
		Number per 0.01 ml.	Percent
6	A	1800	69.3
	B	1750	60.3
12	B	430	14.8
	B	420	14.5
18	A	180	6.93
	B	200	6.81
24	B	24	0.827
	B	29	1.00
30	A	8.5	0.327
	B	4.5	0.155
36	B	0.75	0.026
	B	0.50	0.017
42	B	0.50	0.017

[a] Mean of 10 simultaneous determinations.

The equation of the rate-of-destruction curve of spores of *B. stearothermophilus* No. 1518 in phosphate buffer (pH 7.0) at 131.5° C. (*268.7°* F.) was found to be:

$$\bar{Y}_X = 0.0864 - 0.1089\ (X - 22.62)$$

where $\bar{Y}_X$ is the estimate of the mean Y when X is given, $Y = \log_{10}$ % surviving organisms, and X = time in bath in seconds *(2)*. The *99.7%* confidence limits of the D of the curve are 0.147 minute (*8.82* seconds) and 0.160 minute (*9.60* seconds), where the most probable D is 0.153 minute (9.18 seconds).

Figure 3 shows the regression curve on a scatter diagram of the experimental data.

No corrections for the heating and cooling lags are presented. The lag correction factor (j) for the system is 1.62 and the reciprocal of the slope of the heat penetration curve (f_h) is 2.58 seconds. The times for the center of the infinite cylinder to reach 131.0° C. (*267.8°* F.) and 131.4° C. (*268.5°* F.) have been calculated to be 6.48 and 8.31 seconds, respectively. Correction factors could be determined, if desired, by methods mentioned previously in this paper.

The time required for one person to heat-treat, clean, crush, and plate a series of 10 sample tubes and one control tube is approximately one hour. In our laboratory, the spore suspension is prepared, 100 to 150 capillary tubes are filled and sealed, and 10 to 15 series of 10 capillary tubes each are heat-treated, cleaned, and placed in the culture tubes during one day. The sample capillaries in the culture tubes are stored overnight in a refrigerator, and all plating is done on the following day. With *B. stearothermophilus* No. 1518, no decrease in surviving organisms has been noted during the overnight storage in the refrigerator.

SUMMARY

A relatively simple apparatus for the determination of the thermal resistance of bacteria and bacterial spores is described. The apparatus treats ten or more samples simultaneously, and all samples subjected to a particular bath temperature receive identical heating and cooling treatments.

The apparatus can be used over a wide range of temperatures (from below 100° C. to 140° C.—212° F. to 284° F.). It is comparatively inexpensive, as it can be built at a cost of about $100, exclusive of the temperature control devices.

A bacteriological procedure is described that permits determinations of destruction rates by use of the apparatus. In a relatively short time, sufficient data can be accumulated to justify statistical analysis and establishment of the confidence limits of the slopes (or D's) of the rate-of-destruction curves.

Data typical of results obtained with the use of the apparatus and the bacteriological procedure described in this paper are presented. The D of the rate-of-destruction curve of *B. stearothermophilus* No. 1518 at 131.5° C. (*268.7°* F.) has been found to be 0.153 minute (9.18 seconds), with *99.7%* confidence limits of 0.147 minute (*8.82* seconds) and 0.160 minute (*9.60* seconds).

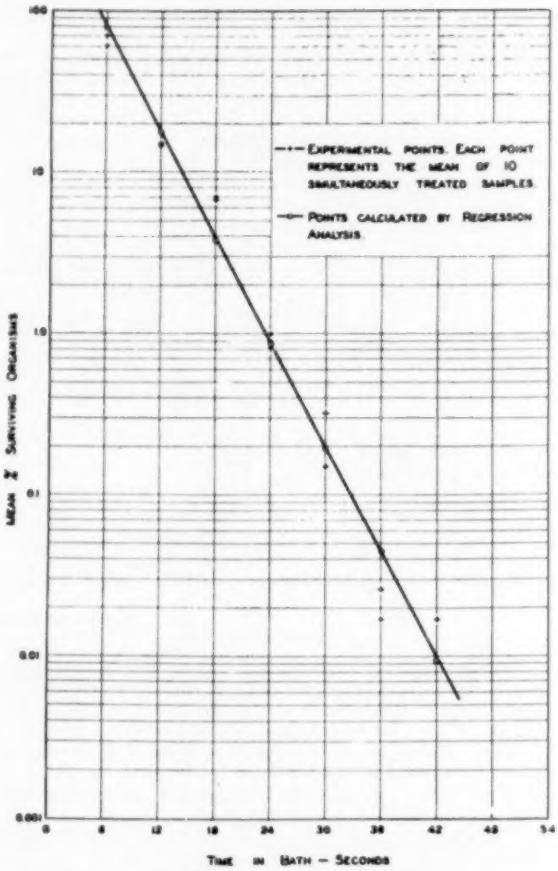
EXPERIMENTAL POINTS. EACH POINT REPRESENTS THE MEAN OF 10 SIMULTANEOUSLY TREATED SAMPLES.
POINTS CALCULATED BY REGRESSION ANALYSIS.
MEAN % SURVIVING ORGANISMS
TIME IN BATH — SECONDS

Figure 3. Regression curve on a scatter diagram of the experimental data obtained with samples of B. stearothermophilus No. 1518, showing the relation between the mean percentage of surviving organisms and the time the samples were heat-treated at 131.5° C. (268.7° F.).

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Retention of Quality in Dehydrated Vegetables Through In-Package Desiccation

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A comprehensive study of the effect of in-package desiccation on the storage stability of dehydrated white potato, sweet potato, cabbage, carrot, beet, and onion is reported. It was demonstrated that substantial protection against nonenzymatic browning can be obtained by the in-package desiccation of these products. The losses of ascorbic acid and of sulfite were also markedly reduced. Oxygen absorption was reduced at 120° F. (49° C.) but was accelerated at 75° F. (24° C.). Packed in air, accelerated oxidative changes (fading and off-flavor development) were observed with sweet potato but not in this test with the other vegetables, thus suggesting that for some products, nitrogen- or vacuum-packing may prove desirable in conjunction with in-package desiccation. Since the temperature coefficient of desiccation (2- to 3-fold per 18° F. or 10° C.) is much lower than that of browning (5- to 8-fold per 18° F.), best protection from in-package desiccation will result if the packaged product is held at moderate temperature until a substantial part of the desiccation has occurred.

Early in the course of development work on dehydrated vegetables for use in World War II, it became evident that lowering of the moisture level is one of the most effective means of decreasing the tendency of these products to brown whenever high storage temperatures were encountered in the field. Nonenzymatic browning (usually called browning) was recognized as the principal type of deterioration occurring under these conditions. In view of this situation, specification moisture contents for specific dehydrated vegetables were fixed by the U. S. Army Quartermaster Corps at the lowest level consistent with current commercial feasibility. Nevertheless, laboratory findings indicated that specification moisture levels for most of the dried vegetables should be greatly lowered for satisfactory protection against browning of these products in military storages and transports. Practical achievement of the desired low moisture levels is difficult because of the low equilibrium relative humidity and slow rate of moisture diffusion in vegetable tissue in this moisture range, especially at the low temperatures required to avoid heat damage during drying.

One method of attaining ultra-low moisture levels involves use of a desiccant in the package of dehydrated vegetables. In this process, called in-package desiccation (IPD), drying takes place during storage through gradual transfer of moisture from product to desiccant. To avoid contamination of product, the desiccant is held in a moisture-permeable, sift-proof container.[c] Other

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[c] The use of desiccant-packaging with other products has been described by a number of investigators, beginning with Ohlhaver (*15, 16, 17*). No attempt to review this literature is made in this paper.

methods of lowering moisture content below specification levels include vacuum desiccation and bin drying with desiccated air. When in-package desiccation of dehydrated vegetables was first announced by Howard *(7, 8)* in 1945, it appeared to be the most promising of these three methods from the standpoint of ready adoption, with due consideration for availability of supplies, equipment, know-how, and feasibility of achieving ultra-low moisture levels within a reasonable time. As the situation relative to these considerations is much the same today, detailed information on method and effectiveness of IPD appears timely. Lovett *(13)* has briefly described cooperative work by the J. R. Simplot Co., Caldwell, Idaho, and the Filtrol Corp., Los Angeles, confirming the protection against browning obtainable by this process with dehydrated onion flakes, and demonstrating that the caking of dehydrated onion powder can be prevented by this method.

This paper is concerned with the quantitative evaluation of the effectiveness of IPD as an aid in maintaining original quality in dehydrated cabbage, onion, carrot, beet, sweet potato, and white potato stored at various temperatures in air and nitrogen atmospheres. Further information on various phases of IPD will be published in forthcoming papers *(3, 19)*. A companion article *(6)* pertains to the rate of desiccation in the samples used in this study. These papers are part of a series *(9, 10, 11)* describing results of extensive storage experiments on dehydrated vegetables at this Laboratory during World War II.

EXPERIMENTAL

Design of experiment. Comparative storage tests were made at various temperatures on samples of dehydrated vegetables packaged with and without desiccant. Half the series of samples were packed in air; the other half were in nitrogen containing about 1.5% oxygen. Packages consisted of sanitary tinned cans—401 x 411 for cabbage and 211 x 414 for other dehydrated vegetables. About 12 cans of product were provided for each experimental condition. At periodic intervals, packages were withdrawn from storage for analysis and appraisal of quality.

Experimental material. The dehydrated vegetables were obtained from commercial sources. In the case of white potato and carrot, sulfited and non-sulfited samples were derived from the same lots of raw material. All products were prepared and packaged in accordance with specifications of the U. S. Army Quartermaster Corps *(20)*. Upon arrival at this Laboratory, the several packages of each category were emptied into a large container, and the product was mixed and allowed to stand in the closed container about 2 weeks at 24° C. (75° F.) for moisture equilibration. Pertinent data on composition of experimental materials are listed in Table 1.

Desiccant and desiccant container. The activated lime used as desiccant in these experiments was prepared from commercial lime. The procedure involved slaking followed by calcining at about 600° C. (1112° F.). The calcined lime was packaged in small tinned (ointment) boxes equipped with perforated tops to allow passage of moisture. Either filter paper or cloth was placed under the lid to prevent sifting of lime into the product. A varied amount of lime was used, depending on extent of desiccation desired. For white and sweet potato this amounted to 21% of the weight of the vegetable. For beet the rate of addition was about 14%, and for the other vegetables it was about 10%.

Methods of appraisal. Methods used for analysis of sulfur dioxide *(18)*, moisture *(14)*, sugars *(21)*, ascorbic acid *(12)*, water soluble (brown) color *(4)*, and carbon dioxide and oxygen *(9)* have been described in previous papers. Moisture content is reported on a moisture-free basis (MFB). It is to be noted that the cited moisture method (with the exception of cabbage and onion) yields values higher (about 1.5 to 2%, absolute) than those obtained by the so-called 6-hour method described in Tentative Specifications, U. S. Army Quartermaster Corps *(20)*. In the case of dried beets, the water soluble color method *(4)* was modified so that measurements reflected changes in natural pigment. Gradual conversion of the pigment to brown color is recognized as a major deteriorative change occurring in dried beets stored at elevated temperatures. The method used for beets is as follows: Dehydrated beets are ground through an 8-mesh screen. A 5-g. sample (MFB) is then placed in a 250-ml. flask with 200 ml. distilled water. The flask, glass-stoppered, is shaken for 2 hours. Its contents are then poured into a funnel equipped with an 18.5 cm. Whatman No. 3 filter paper. The filtrate collected in the first 15 minutes is returned to the funnel for a second pass. As soon as sufficient clear filtrate is collected, 10 ml. is diluted to 250 ml. The percent transmission of the diluted extract is determined in a photoelectric colorimeter, using a 515 mμ color filter and a 2-cm. cell. The optical density is recorded under $E^{0.1\%}_{2\text{cm.},\ 515\ m\mu}$.

TABLE 1
General information and analytical data on dehydrated vegetables used for storage studies

Commodity [a]	Sulfur dioxide (MFB)	Moisture (MFB) [b]	Sugar as glucose (MFB) total	Sugar as glucose (MFB) reducing	Ascorbic acid (MFB)	Carotene (MFB)
	p.p.m.	*%*	*%*	*%*	*mg./100 gm.*	*mg./100 gm.*
White potato	0	8.0	2.2	1.3	28	—
	400	8.0	2.6	1.4	33	—
Carrot	0	6.0	49.5	10.5	—	94
	660	6.0	49.2	8.0	—	96
Cabbage	930	3.2	48.4	47.0	293	—
Onion	0	3.6	73.0	40.4	29	—
Beet	0	6.4	63.6	2.5	—	—
Sweet potato	0	7.4	37.3	26.8	—	—

[a] Product form was as follows: carrot, sweet potato and beet were full dice (⅜ x ⅜ x ⅝ inch before dehydration); white potato was half dice (⅜ x ⅜ x 5/16 inch); cabbage was in shreds; onion was in flakes.
[b] MFB indicates moisture-free basis.

The large number of samples involved in this study necessitated a simplified procedure for subjective appraisal. The taste panel (5 members) was therefore used only to indicate whether samples were satisfactory or unsatisfactory in appearance, flavor and texture. Majority opinions on 2 successive tastings were required for conclusions in this regard. Estimation of degree of browning was based on water-soluble color measurements as described in previous papers *(4, 9, 11)*.

Treatment of data. Results of objective measurement of the brown color formed during storage have been expressed as time intervals required for development of 3 respective degrees of browning. This procedure was described in a previous report *(11)*. The lowest degree (Low) is in the neighborhood of a barely visible change (in appearance of the cooked product). The highest degree (High) represents 5 times as much browning, on the basis of "soluble color" measurement. This would correspond roughly to the "limit of palatability" defined in a previous paper *(9)*. The other level (Medium) is midway between Low and High.

The values for browning tabulated in Tables 2 to 7 were read from plots of optical density versus time as shown in Figures 1 and 2. These illustrative browning curves were drawn by inspection through experimental points.

Sulfite data have been reported as time required for loss of half the original concentration (half life). This treatment of data is based on results of a previous study *(10)* which indicated that disappearance of sulfite in dehydrated vegetables can be approximately represented as a first-order reaction.

Oxygen concentrations in the sample containers as measured through use of the Orsat gas analysis apparatus have been plotted against time in storage. From these plots, the time required for 100 g. of vegetable (moisture-free basis) to absorb designated amounts of oxygen has been calculated and reported in Tables 2 to 7.

TABLE 2

Dried white potato. Effect of in-package desiccation (IPD) on browning, ascorbic acid retention, sulfite loss, and oxygen absorption at various temperatures

Temp. °C.	Pkg. atmos.	SO_2 (initial) (MFB) P.P.M.	IPD [a]	H_2O [b] (final) (MFB) %	Browning Days for specified degrees [c]			SO_2 Half-life days	Oxygen Days for absorption of specified volume (ml.)[d]		Ascorbic acid retention in specified time		Organoleptic appraisal [e]		
					Low	Med.	High		5	10	Days	%	Appearance	Flavor	Texture
49	N_2	400	+	1.4	20	50	>63	>63	—	—	15	100	50-63	50-63	50-63
49	N_2	400	−	8.0	8	16	21	13	—	—	15	100	15-22	15-22	15-22
49	Air	400	+	1.4	22	—	>63	>63	16	50	15	92	50-63	50-63	>63
49	Air	400	−	8.0	8	—	21	13	14	>29	15	75	12-22	12-22	6-12
49	N_2	0	+	1.4	10	33	56	—	—	—	15	100	11-16	11-16	41-57
49	N_2	0	−	8.0	2	4	10	—	—	—	15	100	4-9	4-9	11-16
49	Air	0	+	1.4	10	32	54	—	9.5	>56	15	93	11-16	16-23	23-44
49	Air	0	−	8.0	2	4	10	—	5.2	>16	15	57	4-9	4-9	11-16
38	N_2	400	+	1.6	340	500	720	500	—	—	150	97	482-531	531-623	>623
38	N_2	400	−	8.0	63	135	177	90	—	—	150	89	134-174	174-202	174-202
38	Air	400	+	1.6	250	510	770	480	30	90	150	75	531-623	328-475	>623
38	Air	400	−	8.0	55	120	155	58	40	130	150	40	134-202	134-202	>202
38	N_2	0	+	1.6	95	280	480	—	—	—	150	97	266-322	203-266	322-427
38	N_2	0	−	8.0	18	54	91	—	—	—	150	85	22-52	52-79	107-135
38	Air	0	+	1.6	100	305	510	—	30	90	150	78	266-322	266-322	322-427
38	Air	0	−	8.0	17	50	83	—	30	90	150	32	22-52	79-107	107-135
24	N_2	400	+	2.4	643	—	—	>643	—	—	600	91	>643	>643	>643
24	N_2	400	−	8.0	643	—	—	>643	—	—	600	78	>643	>643	>643
24	Air	400	+	2.4	643	—	—	>643	70	210	600	47	>643	>643	>643
24	Air	400	−	8.0	643	—	—	500	100	370	600	13	>643	>643	>643
24	N_2	0	+	2.4	643	—	>644	—	—	—	600	85	>644	>644	>644
24	N_2	0	−	8.0	280	—	>644	—	—	—	600	50	>644	>644	>644
24	Air	0	+	2.4	>644	—	>644	—	45	200	600	40	>644	>644	>644
24	Air	0	−	8.0	280	—	>644	—	60	300	600	14	>644	>644	>644

[a] IPD samples held 25 days at 24° C. (75° F.) prior to transfer to specified storage temperature. Moisture content at time of transfer: 5.0 percent.

[b] Refers to moisture content at longest time indicated under Browning.

[c] L. M. H. represent changes in optical density ($\Delta E^{1\%}_{2cm.}$, 390 mμ) of 0.035, 0.105 and 0.175 units respectively.

[d] On basis of ml. (NTP) per 100 gm. potato (MFB).

[e] First number = longest number of days when sample was scored acceptable. Second number = shortest number for unacceptable score.

Results of periodic assays for ascorbic acid were plotted against storage time. The percent ascorbic acid retained at the end of a specified period of storage was obtained from the plots and recorded in Tables 2 to 7.

The values recorded under Organoleptic Appraisal roughly indicate the storage interval over which the sample remained satisfactory, in the opinion of the taste panel, with regard to the designated characteristics. Thus, the first entry (50-63) under appearance (Table 2) signifies that at the end of 50 days' storage, under the stated conditions, the sample was found satisfactory, whereas after 63 days (next test period) another sample of the series was voted unsatisfactory.

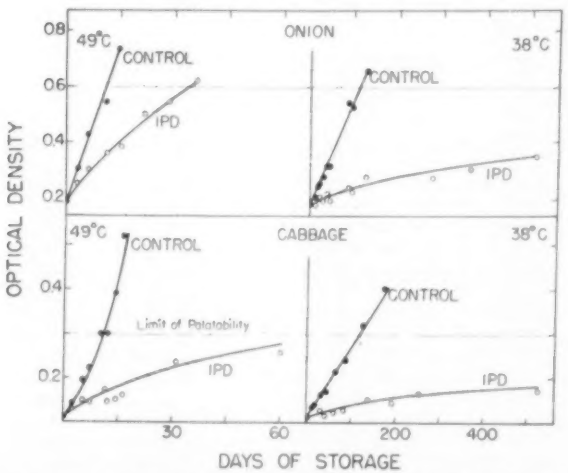

Figure 1. Effect of in-package desiccation (IPD) on the nonenzymatic browning of nitrogen-packed dehydrated onion and cabbage (sulfited), initial moisture contents 3.6 and 3.2%, MFB, respectively. Initial SO_2 content of the cabbage, 930 ppm, MFB.

RESULTS AND DISCUSSION

Moisture change during storage. The possibility of increase in moisture content during storage of dehydrated vegetables at elevated temperatures has long been recognized. This would be expected where severe browning occurs; however, for samples in the palatable range the effect is apparently too small to detect by ordinary methods. Dehydrated onion appears to be an exception to this generalization as indicated by data in Table 4, which show a consistent increase in moisture content of samples stored at 100° F. (38° C.). While the change is not large, it could materially increase the rate of browning (9).

Retardation of browning. Results summarized in Tables 2, 3, 4, 5, and 7 show that browning of dehydrated vegetables was

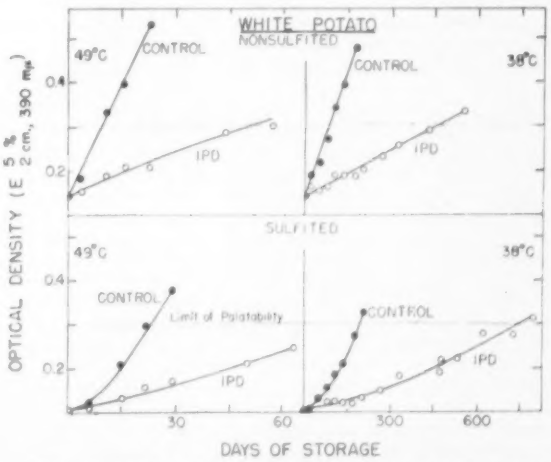

Figure 2. Effect of in-package desiccation (IPD) on the nonenzymatic browning of nitrogen-packed dehydrated white potato. Initial moisture content, 8.0%, MFB. Initial SO_2 content of sulfited samples, 400 ppm, MFB.

TABLE 3

Dried sulfited cabbage.[a] Effect of in-package desiccation (IPD) on browning, ascorbic acid retention, sulfite loss, and oxygen absorption at various temperatures

Temp. ° C.	Pkg. atmos.	IPD[b]	H_2O[c] (final) (MFB) %	Browning Days for specified degrees[d]			SO_2 Half-life days	Oxygen Days for absorption of specified volume (ml.)[e]		Ascorbic acid retention in specified time		Organoleptic appraisal[f]		
				Low	Med.	High		10	20	Days	%	Appearance	Flavor	Texture
49	N_2	+	0.4	7	36	>60	>60	—	—	14	83	>60	>60	>60
49	N_2	—	3.2	3	8	11	15	—	—	14	51	10-12	14-16	14-16
49	Air	+	0.4	6	31	>60	>60	13	44	14	63	42-60	42-60	42-60
49	Air	—	3.2	2.6	7	9	8	4.5	10	14	24	7-10	7-10	>14
38	N_2	+	0.7	180	>525	—	>525	—	—	120	87	>525	357-469	>525
38	N_2	—	3.2	24	74	120	165	—	—	120	54	84-127	84-127	>127
38	Air	+	0.7	180	>525	—	>525	45	100	120	76	>525	357-469	>525
38	Air	—	3.2	21	64	100	95	15	45	120	23	84-127	84-127	>127
24	N_2	+	1.2	>670	—	—	>511	—	—	500	87	>670	>670	>670
24	N_2	—	3.2	>670	—	—	>511	—	—	500	79	>670	>670	>670
24	Air	+	1.2	>670	—	—	>511	50	85	500	71	>670	>670	>670
24	Air	—	3.2	>670	—	—	>511	150	350	500	36	>670	>670	>670

[a] Sulfite content (initial) 930 P.P.M. SO_2.

[b] IPD samples held 21 days at 24° C. (75° F.) prior to transfer to specified storage temperature. Moisture content at time of transfer, 2.6 percent.

[c] (Final) refers to moisture content at longest time indicated under Browning.

[d] L. M. H. represent changes in optical density ($\Delta E^{2.5\%}_{1\,cm.}$, 420 mμ) of 0.040, 0.120, and 0.200 units respectively.

[e] On basis of ml. (NTP) per 100 gm. cabbage (MFB).

[f] First number = longest number of days when sample was scored acceptable. Second number = shortest number for unacceptable score.

TABLE 4

Dried onion. Effect of in-package desiccation (IPD) on browning, ascorbic acid retention and oxygen absorption at various temperatures

Temp. ° C.	Pkg. atmos.	IPD[a]	H_2O (MFB) %		Days for specified degrees[d]			Oxygen Days for absorption of specified volume (ml.)[e]		Ascorbic acid retention in specified time		Organoleptic appraisal[f]		
			I[b]	F[c]	Low	Med.	High	10	20	Days	%	Appearance	Flavor	Texture
49	N_2	+	2.8	1.3	4	17	32	—	—	15	96	22-29	22-29	>43
49	N_2	—	3.6	4.0	2.2	7	11	—	—	15	50	10-15	>15	>15
49	Air	+	2.8	1.1	3	14	37	35	>43	15	79	22-29	22-29	>43
49	Air	—	3.6	4.0	1.3	3.9	6.5	10	>15	15	41	6-10	6-10	>15
38	N_2	+	2.8	1.0	190	>515	—	—	—	180	100	>486	>486	>486
38	N_2	—	3.6	4.0	24	72	120	—	—	180	90	88-98	88-98	>189
38	Air	+	2.8	1.1	230	>515	—	130	400	180	96	366-486	>486	>486
38	Air	—	3.6	4.0	25	75	125	80	170	180	60	88-98	88-98	>189
24	N_2	+	2.8	1.2	>560	—	—	—	—	650	98	>673	>673	>673
24	N_2	—	3.6	3.6	470	—	—	—	—	650	83	>673	>673	>673
24	Air	+	2.8	1.2	>560	—	—	310	>680	650	95	>673	>673	>673
24	Air	—	3.6	3.6	480	—	—	650	>680	650	60	>673	>673	>673

[a] IPD samples held 23 days at 24° C. (75° F.) prior to transfer to specified storage temperature.

[b] At beginning of storage at indicated temperature.

[c] Refers to moisture content at longest time indicated under Browning.

[d] L. M. H. represent changes in optical density ($\Delta E^{2\%}_{1\,cm.}$, 390 mμ) of 0.080, 0.240, and 0.400 units respectively.

[e] On basis of ml. (N. T. P.) per 100 gm. Onion (MFB).

[f] First number = longest number of days when sample was scored acceptable. Second number = shortest number for unacceptable score.

greatly retarded through use of in-package-desiccation (IPD). In all instances the control samples browned more rapidly at 120° F. (49° C.) and 100° F. (38° C.) than the corresponding IPD samples. As the latter temperature is presented emphasized in stability tests on dehydrated vegetables, it should be of interest to compare time intervals required for development of a low degree of browning at 100° F. (38° C.) in paired IPD and control samples. The ratio of the time intervals, IPD/control, indicates the advantage that might be derived through in-package desiccation. Of course, the magnitude of advantage would vary depending in large measure on the respective moisture levels of the IPD and control samples. For the nitrogen-packed vegetables these ratios are roughly 8:1 for onion and sulfited cabbage; 5:1 for sulfited and non-sulfited white potato; and 2:1 for sulfited and non-sulfited carrot. Ratios of similar magnitude were obtained for the air-packed samples of these vegetables. It is evident that the greatest advantage of IPD was found with cabbage and onion while the least advantage was with carrot. Intermediate results were obtained on in-package desiccation of white potato. The relatively slow rate of desiccation of carrot and the relatively high terminal moisture content of IPD samples account for the small advantage obtained with this product.

It is noteworthy that the IPD samples of onion, sulfited cabbage, and sulfited white potato suffered only a low degree of browning after 6 months' storage at 100° F. (38° C.). These results indicate that browning of these products at elevated temperatures could be satisfactorily retarded through use of IPD for onion and of IPD in combination with sulfite (judiciously used) for white potato and cabbage. Browning of the sweet potato was very slow, even for the samples without desiccant, suggesting that IPD may be unnecessary with this product.

In the case of dehydrated beet (Table 6) IPD proved quite effective for retarding deterioration of the red beet pigment at elevated temperatures. While the nitrogen-packed samples, stored at 100° F. (38° C.), suffered a low degree of change in natural color in 120 days, as compared to 50 days for the control samples, further change in color of the IPD samples was very slow. After 610 days less than a medium degree of color change had taken place in the IPD samples, whereas this degree of discoloration was evident in the control samples after 140 days.

TABLE 5

Dried carrot. Effect of in-package desiccation (IPD) on browning, sulfite loss, and oxygen absorption at various temperatures

Temp. ° C.	Pkg. atmos.	SO_2 (initial) (MFB) P.P.M.	IPD [a]	H_2O [b] (final) (MFB) %	Browning Days for specified degrees [c]			SO_2 Half-life days.	Oxygen Days for absorption of specified volume (ml.) [d]		Organoleptic appaisal [e]		
					Low	Med.	High		5	10	Appearance	Flavor	Texture
49	N_2	660	+	3.7	12	32	46	17	—	—	41-48	41-48	>48
49	N_2	660	—	6.0	8	21	31	9	—	—	13-20	20-27	34-41
49	N_2	0	+	3.7	6	16	27	—	—	—	27-34	20-27	34-41
49	N_2	0	—	6.0	4	12	20	—	—	—	27-34	7-13	34-41
49 [f]	N_2	660	+	3.7	15	33	>40	—	—	—	—	—	—
49 [f]	N_2	660	—	6.0	7	19	28	—	—	—	—	—	—
49 [f]	Air	660	+	3.7	14	30	>40	—	25	>40	—	—	—
49 [f]	Air	660	—	6.0	7	19	28	—	16	35	—	—	—
49 [f]	N_2	0	+	3.7	9	25	>36	—	—	—	—	—	—
49 [f]	N_2	0	—	6.0	4	12	20	—	—	—	—	—	—
49 [f]	Air	0	+	3.7	9	24	>36	—	25	>36	—	—	—
49 [f]	Air	0	—	6.0	4	12	20	—	10	22	—	—	—
38	N_2	660	+	3.9	102	>275	>275	100	—	—	247-275	184-247	>275
38	N_2	660	—	6.0	45	135	>184	63	—	—	156-184	>184	>184
38	Air	660	+	3.9	95	>184	—	80	23	50	0-100	100-121	>184
38	Air	660	—	6.0	42	125	>184	30	22	45	0-100	100-121	>184
38	N_2	0	+	3.9	53	160	>175	—	—	—	121-156	121-156	>175
38	N_2	0	—	6.0	26	79	130	—	—	—	27-100	121-156	156-175
38	Air	0	+	3.9	57	170	>175	—	20	45	0-100	121-175	>175
38	Air	0	—	6.0	25	75	125	—	25	55	0-100	0-100	121-175
24	N_2	660	+	4.3	>625	—	—	>625	—	—	>625	>625	>625
24	N_2	660	—	6.0	>625	—	—	>625	—	—	>625	>625	>625
24	Air	660	+	4.3	>625	—	—	>625	28	65	163-457	163-457	>625
24	Air	660	—	6.0	>625	—	—	500	31	80	163-457	163-457	457-625
24	N_2	0	+	4.3	>625	—	—	—	—	—	>457	>457	>457
24	N_2	0	—	6.0	450	—	—	—	—	—	>457	>457	>457
24	Air	0	+	4.3	>625	—	—	—	27	64	107-163	163-457	>457
24	Air	0	—	6.0	420	—	—	—	29	75	20-107	107-163	>457

[a] IPD samples held 23 days at 24° C. (75° F.) before transfer to specified storage temperature. Moisture content at time of transfer, 5.3 percent, MFB.

[b] At end of storage at indicated temperature.

[c] Low, Medium, and High represent optical density changes ($\Delta E^{1.8\%}_{1\,cm}$, 420 mμ) of 0.060, 0.180, and 0.300 units respectively.

[d] On basis of ml. (N. T. P.) per 100 gm. carrot (MFB).

[e] First number = longest number of days when sample was scored acceptable. Second number = shortest number for unacceptable score.

[f] Samples stored 65 days at 38° C. (100° F.) (after 23 days' initial storage at 24° C.) before transfer to storage 49° C. (120° F.). Moisture content at time of transfer, 4.5 percent, MFB.

TABLE 6

Dried beet. Effect of in-package desiccation (IPD) on retention of natural color and oxygen absorption at various temperatures

Temp. ° C.	Pkg. atmos.	IPD [a]	H_2O [b] (final) (MFB) %	Color Days for specified degrees [c] of change			Oxygen Days for absorption of specified volume (ml.) [d]		Organoleptic appaisal [e]		
				Low	Med.	High	5	10	Appearance	Flavor	Texture
49 [f]	N_2	+	3.0	50	>97	—	—	—	—	—	—
49 [f]	N_2	—	6.4	8	22	33	—	—	—	—	—
49 [f]	Air	+	3.0	65	>97	—	60	>82	—	—	—
49 [f]	Air	—	6.4	8	22	33	12	22	—	—	—
49	N_2	+	3.0	35	>95	—	—	—	>95	>95	>95
49	N_2	—	6.4	14	27	36	—	—	23-36	23-36	23-36
49	Air	+	3.0	35	>95	—	42	95	>95	>95	>95
49	Air	—	6.4	12	23	29	12	18	15-30	15-30	36-49
38	N_2	+	3.3	120	>610	—	—	—	>610	522-610	>610
38	N_2	—	6.4	50	140	210	—	—	151-340	151-340	151-340
38	Air	+	3.3	90	>610	—	130	290	>610	522-609	>610
38	Air	—	6.4	50	130	180	50	85	116-151	151-249	>249
24	N_2	+	4.4	>627	—	—	—	—	>627	>627	>627
24	N_2	—	6.4	>627	—	—	—	—	>627	459-627	>627
24	Air	+	4.4	>627	—	—	140	350	>627	459-627	>627
24	Air	—	6.4	>627	—	—	200	350	>627	459-627	>627

[a] IPD samples held 21 days at 24° C. (75° F.) before transfer to specified temperatures. Moisture content at time of transfer, 5.8 percent, MFB.

[b] At end of storage at indicated temperature.

[c] Low, Medium, and High represent optical density changes ($\Delta E^{3.1\%}_{2\,cm}$, 515 mμ) of 0.050, 0.150 and 0.250 units respectively.

[d] On basis of ml. (N. T. P.) per 100 gm. Beet (MFB).

[e] First number = longest number of days when sample was scored acceptable. Second number = shortest number for unacceptable score.

[f] Samples stood 48 days at 38° C. (100° F.) (after 21 days initial storage at 75° F.) before transfer to storage at 49° C. (120° F.). Moisture content at time of transfer, 3.7%, MFB.

TABLE 7

Dried sweet potato. Effect of in-package desiccation (IPD) on browning, ascorbic acid retention, and oxygen absorption at various temperatures

Temp. ° C.	Pkg. atmos.	IPD [a]	H_2O [b] (final) (MFB) %	Browning Days for specified degrees [c]			Oxygen Days for absorption of specified volume (ml.) [d]		Ascorbic acid retention		Organoleptic appraisal [e]		
				Low	Med.	High	10	20	Days	%	Appearance	Flavor	Texture
49	Air	+	3.0	>76	...	...	28	>76	64	66	22-29	64-76	>76
49	Air	−	7.4	14	40	68	9	22	64	11	49-64	49-64	64-76
38	N_2	+	3.0	>557			...	...	....	....	>557	>557	>557
38	N_2	−	7.4	140	420	>557	...	...	....	....	>557	>557	>557
38	Air	+	3.0	>151		...	50	>151	....	....	36-49	49-89	>151
38	Air	−	7.4	120	>151	...	20	56	....	....	89-151	89-151	>151
24	N_2	+	3.2	>527	...	...	...	...	557	100	>557	445-557	>557
24	N_2	−	7.4	>527	...	...	...	...	557	37	>557,	445-557	>557
24	Air	+	3.2	>407	...	...	60	200	407	72	9-49	9-49	>407
24	Air	−	7.4	>407	...	...	90	250	407	28	165-407	165-407	>407

[a] IPD samples stood 20 days at 24° C. (75° F.) before transfer to specified storage temperature.

[b] At end of storage at indicated temperature.

[c] Low, Medium, and High taken as optical density changes ($\Delta E^{2.5\%}_{1\text{ cm.}}$, 420 mμ) of 0.025, 0.075, and 0.125 units respectively.

[d] On basis of ml. (N. T. P.) per 100 gm. sweet potato (MFB).

[e] First number = longest number of days when sample was scored acceptable. Second number = shortest number for unacceptable score.

Although IPD proved effective with dehydrated vegetables stored at 120° F. (49° C.), the advantage was smaller than that observed in samples stored at 100° F. (38° C.). This difference appears reasonable as increasing temperature affects the browning rate more than the rate of in-package desiccation. As previously reported *(9, 11)*, an 18° F. (10° C.) temperature rise leads to approximately 5- to 8-fold increase in browning rate, whereas the rate of IPD was found to increase about 2- to 3-fold *(6)*. In view of these considerations, some samples were held at 100° F. (38° C.) for 1½ to 2 months to allow desiccation to proceed to some extent before exposure to 120° F. (49° C.) storage. For dehydrated beet, this procedure was quite effective. The time ratio, IPD/control, for a low degree of change in color at 120° F. (49° C.) was about 7:1 for the samples delayed 48 days at 100° F. (38° C.), while the ratio was about 3:1 for comparable samples placed directly in storage at the higher temperature. A similar effect was noted in a parallel experiment with dehydrated carrot but the magnitude of difference between the ratios was much smaller.

Retention of sulfite. Data in Tables 2, 3, and 5 show that IPD effectively retarded the rate of disappearance of sulfite present in dehydrated potato, cabbage, and carrot. This is in accord with previous findings *(10)* which showed that the rate of sulfite loss in dehydrated sulfited carrot and white potato increased roughly 2-fold with each moisture content increase of 1.5 to 2%. For reasons stated above, it is interesting to compare half-life periods of sulfite in paired IPD and control samples stored at 100° F. (38° C.). For the nitrogen packed samples, IPD increased the half life periods roughly 6, 3, and 2-fold in white potato, cabbage, and carrot, respectively. The air-packed samples of these vegetables, in the same order, showed half- life increases of roughly 8-, 6-, and 3-fold. As previously mentioned with regard to browning, the magnitude of advantage would vary depending on the respective moisture contents of the IPD and control samples.

Retention of ascorbic acid. It may be noted that IPD markedly improved retention of ascorbic acid (Tables 2, 3, and 4). As dehydrated cabbage is a good source of this nutrient, special attention is called to the data on this product. It appears noteworthy that IPD retarded loss of ascorbic acid not only in the air-packed but also in the nitrogen-packed samples. Moreover, retardation of ascorbic acid loss was found even at 75° F. (24° C.). Data on ascorbic acid retention in white potato and onion also demonstrate the protective action of IPD. It is evident, however, that removal of oxygen as well as IPD is necessary for maximum retention of this vitamin.

Absorption of oxygen. As previous studies *(2, 5)* have shown that lowering of moisture content tends to favor oxygen absorption, it is important to evaluate the effect of IPD in this regard. Data in Tables 2 to 7 show that air-packed IPD samples stored at 120° F. (49° C.) absorbed oxygen at a lower rate than did the control samples, but at 75° F. (24° C.) the situation was reversed. At 100° F. (38° C.), IPD retarded absorption of oxygen by beet, onion, sweet potato, and cabbage, while it accelerated oxygen uptake by sulfited white potato. The effect was very slight, if any, in carrot and non-sulfited white potato. The data do not permit explanation of these results; however, they do suggest that IPD retards oxygen absorption by systems related to browning and accelerates oxygen absorption by other systems.

Retention of organoleptic quality. For purposes of this discussion it appears desirable to distinguish between conditions conducive to browning, where IPD is beneficial, and those where browning is unimportant. For air-packed dehydrated vegetables held at room temperature, 75° F. (24° C.) storage life is commonly limited by oxidative changes, while at temperatures of 100° F. and higher storage life is usually limited by browning. The air-packed sweet potatoes used in this study were an exception to this generalization; their storage life at 100° F. (38° C.) and even to some extent at 120° F. (49° C.) was considered limited by oxidative changes (fading and development of stale or rancid flavor) rather than by browning.

With regard to room-temperature storage, taste panel results for the white potato, cabbage, onion, carrot and beet generally show neither advantage nor disadvantage for the cooked in-package-desiccated samples as compared to their controls. Further study of the effect of IPD on changes in air-packed dehydrated vegetables would be desirable since the method of appraisal was not designed for high precision and since most of these products did not become unacceptable during the storage periods employed. The very long storage period (643 to 673 days) over which the air-packed dehydrated cabbage, white potato, and onion were considered acceptable, and the fact that the IPD samples of air-packed non-sulfited carrot remained acceptable for a longer period than did their controls, suggest that with these vegetables IPD has little if any significant effect on the rate of changes in color and flavor of these products in air at 75° F.

With the air-packed sweet potato, however, fading and development of stale flavor were accelerated by in-package desiccation. For sweet potato this result is perhaps of little importance since the sweet potato was so resistant to browning that IPD appears unnecessary, but it does suggest that for some dehydrated products nitrogen- or vacuum-packing may prove desirable for control of oxidative changes in conjunction with IPD or other method of attaining low moisture for control of browning.

Oxidative changes would of course be controlled by nitrogen-packing; it may be noted that IPD had no apparent effect on the flavor and appearance of the sweet potato stored at room temperature in an atmosphere of nitrogen.

For the storage at 100° F. (38° C.) and 120° F. (49° C.) the organoleptic appraisals of the samples other than the air-packed sweet potato show a marked advantage in favor of the in-package desiccated samples. As would be expected, IPD prolonged retention of satisfactory appearance and flavor. At the same time, it improved retention of satisfactory texture. In these studies, unsatisfactory texture was usually associated with poor reconstitution on cooking.

It is noteworthy that the IPD samples of dehydrated onion, beet, sulfited white potato, and sulfited cabbage remained satisfactory in appearance, flavor, and texture for more than a year at 100° F. (38° C.). Dehydrated carrot, sulfited and packed in nitrogen, remained satisfactory for about 6 months at this temperature. Doubtless, a procedure of manufacture that would facilitate more extensive desiccation would lead to longer retention of satisfactory quality in dehydrated sulfited carrots. A smaller piece size than the full dice used in these studies (Table 1) should be beneficial in this regard.

SUMMARY AND CONCLUSIONS

A comprehensive study of the effect of in-package desiccation (IPD) on the storage stability of dehydrated white potato, sweet potato, cabbage, carrot, beet, and onion is reported. Emphasis in this study was on control of nonenzymatic browning during warm storage. Results show that a very substantial protection against browning can be obtained by in-package desiccation. Cabbage and onion were best protected, the lengths of time for given degrees of browning at 100° F. (38° C.) being increased about 8-fold by IPD. It is considered significant that, in the opinion of the panel, the IPD samples of sulfited cabbage, sulfited white potato, beet, onion, and nitrogen-packed sweet potato remained satisfactory in appearance, flavor, and texture for more than a year at 100° F. (38° C.).

The retention of ascorbic acid was markedly improved by in-package desiccation of the products tested (cabbage, white potato, sweet potato, and onion). This was true at all temperatures (75, 100, and 120° F.) and in both nitrogen and air.

Disappearance of sulfite was also effectively retarded.

For dehydrated beet, the degradation of the natural red pigment was much reduced. For air-packed sweet potato, however, the fading of natural pigment was accelerated.

For storage at 75° F., in-package desiccation appeared to have little effect on the acceptability of most of these products, whether packed in nitrogen or in air. With sweet potato, however, fading and off-flavor development were accelerated for those samples packed in air, but not those packed in nitrogen. With certain other commodities, packing in nitrogen or in vacuum may therefore be desirable in conjunction with IPD if maximum storage life is to be obtained.

In the case of sweet potato, browning was so low that in-package desiccation appears unnecessary, but the oxidative changes proceeded so fast as to suggest that sweet potato should be packed in an oxygen-free atmosphere.

The protection against browning was smaller at 120° F. (49° C.) than at 100° F. (38° C.). This is considered to be due to the fact that the temperature coefficient of the browning reaction is much higher than that of desiccation. Because of the difference in temperature coefficients, maximum protection against browning will be obtained if the vegetables, packed with a desiccant, are held at a moderate temperature for as long as possible after packaging, to permit as much moisture reduction as possible before exposure to elevated temperature.

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Some Factors Affecting the Whipping Characteristics of Dried Whole Egg Powders[a,b]

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The loss of foam volume in whipping tests with reconstituted whole egg powders is traced to the breakdown of the natural fat emulsion of fresh eggs. This emulsion breakdown causes these powders to act as anti-foam agents and can cause the collapse of a foam produced with fresh eggs. Washing these powders with a fat solvent counters their anti-foam activity and allows them to be whipped to voluminous foams. Further evidence that the free fat is the responsible agent in poor foaming characteristics of dried eggs is shown by photomicrographs illustrating the emulsion breakdown during drying. Some emulsifying agents, such as the alkyl aryl sulfonates, enable the re-formation of the emulsion of fat in water so that such rehydrated products can be whipped. More generally acceptable food emulsifiers are less successful.

The civilian market has not as yet welcomed dried whole egg powders as a substitute for shell eggs in scrambled egg and related dishes. Therefore, at the close of World War II the baking industry became the prime consumer of these powders (exclusive of recurrent military demands). It would be possible with baked goods to use dried eggs having a flavor quality somewhat lower than that required for scrambled egg dishes, because in baked goods off-flavors could be masked to some extent, providing the rehydrated whole egg had the proper functional properties. By functional properties is meant the ability of the product to form a voluminous and sturdy foam when whipped or beaten and, to a lesser degree, the ability to give extensibility through proper coagulation during the baking. The egg should also perform as an emulsifying agent in cakes or baker's products that utilize mixtures of fats and oils with water.

The British investigators *(1)* at the Low Temperature Research Station, Cambridge, England, were apparently among the first to report correlation between a beating test and the baking quality of whole egg powders. Their results on the beating test were expressed in terms of percentage increase in volume, following a standardized beating or whipping procedure. None of the samples of commercially spray-dried powders examined by these investigators gave foam of sufficient volume and stability to allow a direct measurement of foaming power. To form measurable foams it was necessary to add large amounts of sugar—presumably to increase the viscosity. Their test called for the rehydration of 18 g. of whole egg powder, so as to provide 75 g. of rehydrated egg, and the addition of 60 g. of sucrose. Under these conditions they were able to form measurable foams that correlated in volume with the volumes of sponge cakes prepared from the powders. Commercial or stored whole egg powders were always found inferior to those freshly prepared in the laboratory.

In a later modification of this test *(7)*, the beating operation was carried out at 40° C. (104° F.). Beating at this temperature increased the differential between the sample grades and thus gave more accurate comparisons.

The Canadian investigators *(14)* at the National Research Laboratories, Ottawa, Canada, adopted this beating test used by the Low Temperature Research Station with slight modification; they reported a fair correlation with other quality tests such as fluorescence, solubility, and pH. Subsequently they reported a correlation between baking tests (sponge cakes) and foam tests on whole egg powders *(11)*. The procedure for measuring the foam volume varied somewhat from that previously used but still consisted of adding a large amount of sugar and beating at 40° C. (104° F.). Foam volume was measured with a standard deviation of about 2% of the mean and was found to correlate with baking volume within the 1% level of statistical significance.

Pearce and coworkers *(10)* at the National Research Laboratories in Canada and research investigators in England *(4)* reported on the development and subsequent extensive production of a dried egg product called "sugar-dried egg" or "sugar-egg powder." This product was especially developed to make good sponge-type cakes and was produced by mixing sufficient sucrose with the egg melange prior to drying to give a product that, after spray drying, contained two parts of egg solids to one part of sugar solids.

Fryd *(5)* reported that sugar-dried whole egg powder was rather hygroscopic and required special packaging precautions. However, Fryd was generally impressed with the future of sugar-dried eggs on the British market. He described a beating test in which 108 ml. of water at 86° F. (30° C.) was mixed with 54 g. of sugar-dried egg and 100 g. of dried sugar. The mixture was premixed to break up lumps and whipped for ten minutes in a Hobart Beater fitted with an extra bowl for a water jacket of constant temperature. The curve of time of beating *vs.* foam volume was a straight line from 8 to 40 minutes. Fryd also reported that the use of high beating temperatures produced higher foam volumes, but that sponge cakes made from these foams

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were not improved once the limiting figure of about 80° F. (27° C.) was reached.

The pH of a fresh egg is generally about 7.5 prior to drying but changes to about 8.5 during the drying process *(12)*. This indicates the loss of a volatile acid constituent, presumably carbon dioxide. Therefore, at an early date in the period of intensive research on dried whole eggs, stimulated by the rapid expansion of the industry during World War II, it was considered worthwhile to return some of the acid to the egg in the form of a non-volatile acid such as hydrochloric or lactic acid, so that the pH of the reconstituted egg would be about equal to that of the untreated egg. Stewart, Best, and Lowe *(13)* reported that the lowering of the pH in this fashion had a beneficial effect on the solubility of the stored product but did not retard the development of fluorescence. These investigators mentioned that lowering the pH below 6.5 caused a marked alteration in flavor and texture to an extent usually considered objectionable in scrambled egg tests.

Experimentation showed that even better solubility retention in storage was possible if the pH were lowered to 5.5 (the pH of the normal yolk). Such a treatment was organoleptically disqualified until eventual research showed that the dried eggs could be neutralized by mixing dry sodium bicarbonate with the egg powders *(3)*. Thus acidification prior to drying resulted in a product with improved solubility retention in storage and with consequent benefit to organoleptic qualities during storage *(2, 6)*. Since 1949 the U. S. Army purchasing contracts for dried egg powders have specified the acidification step.

This acidification step, however, was designed primarily to improve egg powders intended for consumption as a scrambled egg dish and apparently does not contribute to so great an extent to the retention of quality in baked goods, for example, when the egg powder is used as a sponge cake ingredient. In fact, Lightbody and Fevold *(9)* cite experiments by Conrad (private communication to them) in which there was no beneficial effect of acidification on sponge cake volume either when the egg powder was used alone or in combination with sugar.

Recent developments in dried egg research have shown the benefit of removing the naturally occurring reducing sugars before drying, as an aid in increasing storage life *(8)*. At present this is being done both by yeast fermentation and by treatment with a glucose-oxidizing enzyme. This process has apparently brought about marked progress in stabilizing egg powders during storage.

The review of the pertinent literature as presented by Lightbody and Fevold *(9)* indicates that the major attention of research in the dried egg field appears to have been directed toward the improvement of storage life, and that there has been somewhat less research directed toward solution of the problem of loss of quality during drying. As shown above, a major defect of freshly dried whole egg powder is the loss of foaming or whipping quality. The experiments reported in this present paper, therefore, were designed to investigate the cause of this loss in quality.

EXPERIMENTAL AND RESULTS

Loss of foam volume traced to yolk. It has previously been noted in these laboratories that lyophilized (freeze-dried) egg white shows essentially no loss of foam volume when rehydrated and whipped. On the other hand, lyophilized whole egg melange generally loses one-third or more of its foam volume. Although the addition of large amounts of sugar to rehydrated, lyophilized whole egg will much improve the whipping characteristics, these egg powders are still not the equal of fresh eggs in this respect.

It would seem apparent that the alteration during drying did not occur in the white portion of the egg. This supposition was experimentally investigated by drying the yolk and the white separately and recombining with respective fractions of undried (fresh) eggs. The results of this experiment are shown in Table 1.

TABLE 1

Effect of mixing egg white and yolk on foam volume of rehydrated mixtures

Sample No.	Treatment	Foam specific volume [c]
		cc. per g.
1	Lyophilized egg white	9.5
2	Fresh egg white	9.2
3	Lyophilized whole egg	2.6
4	Fresh whole egg	6.4
5	Lyophilized yolk plus lyophilized white	2.3
6	Fresh white plus lyophilized yolk	2.3
7	Lyophilized white plus fresh yolk	6.1
8	Spray-dried white (fermented and acidified) plus fresh yolk	6.0
9	Spray-dried white (fermented and acidified) plus lyophilized yolk (yolk rehydrated separately)	1.8

[c] No sugar added.

The following generalized conclusions can be drawn from these data:

(a) Dried egg white had essentially the same whipping characteristics as fresh egg white.

(b) A mixture of dried egg white and dried yolk had about the same foam volume as dried whole egg. In either case, the foam volume was much less than the volume produced by fresh whole egg.

(c) A mixture of dried egg white and fresh yolk had a foam volume similar to that of fresh whole egg.

(d) Spray-dried egg white is similar to lyophilized and fresh white.

(e) A mixture of spray-dried egg white and fresh yolk had a foam volume similar to that of fresh whole egg.

(f) A mixture of spray-dried egg white and lyophilized yolk gave a poor volume, which was somewhat similar to that from the lyophilized whole egg.

It is apparent from the above conclusions that the loss of whipping characteristics during drying was associated with the yolk rather than the white of the egg.

Dried whole egg powders as anti-foam agents. Five g. of an old sample of spray-dried whole egg powder (stored about 18 months at room temperature) was added to 50 g. of freshly thawed frozen egg white in a beater bowl and whipped for several minutes. No foam resulted, even though a further experiment indicated that the egg white by itself whipped readily to a voluminous foam. In a third experiment, the egg white was whipped to a sturdy foam before the dried whole egg powder was added. In this case the foam almost immediately collapsed and completely disappeared when the 5 g. of whole egg powder was added to the 50 g. of egg white foam. In repeating the experiment, it was found that it was only necessary to sprinkle a few particles of the dried whole egg on the surface of the whipped egg white to cause a collapse of the foam.

Removal of anti-foam characteristics with acetone. About 50 g. of this stored spray-dried whole egg powder was partially defatted by shaking in 200 ml. of acetone followed by filtration. This washing procedure was repeated 3 times. Most of the bright orange-yellow color was removed, leaving an amber-straw color. The acetone was evaporated by allowing the egg

powder to remain exposed to the laboratory air overnight on an aluminum sheet. No odor of acetone remained after this procedure. Five g. of this partially defatted whole egg powder was mixed with 50 g. of fresh egg white and 15 ml. of water in a mixing bowl. This mixture readily whipped to a foam about equal in volume to that of the fresh egg white. The defatted whole egg powder did not completely dissolve and left some specks of solid material in the foam. It was subsequently demonstrated, however, that this loss of solubility occurred during the storage of the egg powder rather than because of the acetone washing treatment.

When 5 g. of this partially defatted whole egg powder was added to 50 g. of fresh egg white foam, there was no evidence of any collapse of the foam, even with extended beating.

The whipping characteristics of the partially defatted whole egg powder were compared with those of the untreated powder. Both powders were relatively insoluble, and, therefore, did not readily produce voluminous foams. However, the partially defatted powder represented a decided improvement over the untreated powder in this respect and produced a foam volume several times greater than that of the untreated sample.

Subsequent experiments with freshly dried whole egg powders subjected to this acetone washing treatment showed no loss of solubility, and such partially defatted powders when rehydrated whipped to voluminous and sturdy foams.

Also freshly dried whole egg powders exhibited the aforementioned anti-foam characteristics to an extensive degree, although slightly less so than the stored samples.

The acetone was allowed to evaporate from the extract of these whole egg samples. This resulted in a dark yellow, viscous oil at room temperature. A few drops of this oily material added to an egg white foam caused a rapid collapse of the foam. A few drops of acetone added similarly had no effect.

It appears to be fairly conclusive that the anti-foam action of dried whole eggs is associated with the free fat of the powders. About 45% of the dried whole egg is composed of lipid material. It is highly probable that a large portion of this lipid material is in the form of an oily film on the surface of the dry particles.

Microscopic evidence of emulsion breakdown. A microscopic examination of a drying film of whole egg gives a visible record of the transition of the fat from spherical globules within the aqueous phase (i.e., oil droplets in an oil-in-water emulsion) to an uneven film of oil on the surface of the dry whole egg powder. The transition is similar to, although slightly more spectacular than, that occurring with a drying film of egg yolk. Photomicrographs of a film of egg yolk drying at room temperature on a microscope slide are shown in Figure 1.

Early attempts were made to stain the oil globules with fat solvent dyes, such as Sudan III or Sudan IV, or with osmic acid. However, these staining procedures produced artificial conditions, and after some experimentation it was found that the breakdown of the emulsion during drying could be followed without the aid of stains or fixatives.

The procedure used was to place a drop of fresh egg yolk or whole egg melange on a glass slide and allow it to spread to a fairly thick film. The slide was then examined at 150 power by use of a strong spot light focussed to about the area covered by the objective. This revealed discrete oil globules of widely varying diameters from about 10 μ down to a fraction of a micron, with the greater proportion generally between 1 and 5 μ in diameter. Figure 1-A shows these oil globules in the fresh egg yolk. Oil globules are easily distinguished from air bubbles, if present, as the latter are black and non-transparent.

As the water starts to evaporate from the film of egg yolk, the oil globules begin to show a distortion from the perfect spheroid shape and generally take an ellipsoid or flattened shape. This is evident in some of the globules shown in Figure 1-B.

When sufficient water has evaporated so that the water remaining can no longer form a continuous, free phase in which the oil globules can remain suspended, a rather sudden and complete change takes place in the oil phase. The oil globules completely disappear, and the oil then appears as an exudation of extremely small droplets, which dot the surface of the drying film of yolk. This is shown in Figure 1-C. This photomicrograph is reproduced at the same scale of enlargement as the previous figures showing the oil globules in the aqueous phase. Therefore, it is apparent that these pin-point exudations of fat on the drying surface are considerably smaller than the original oil globules in the fresh yolk.

These oil droplets rapidly coalesce to larger, irregularly shaped globules of oil on the surface of the dry film, as is apparent in Figure 1-D. The oil can be seen to run together and coalesce into larger "puddles" on the surface as drying becomes nearly complete. The oil eventually appears as a collection of fairly large agglomerates (Figure 1-E) and at this stage resembles a discontinuous film of water formed on a greasy surface. Apparently the surface tension of the oil causes it to remain as irregular sections of an oily film on the surface of the dry film of the egg yolk.

Figure 1-F shows several of the previously described phases of drying taken in the one exposure. In this case the objective was focussed near an edge of a drying film of egg yolk on a microscope slide. The outside edge is at the left of the photomicrograph, and the degree of drying is at progressively advanced stages following across the photomicrograph.

This microscopic evidence of emulsion breakdown during drying substantiates physico-chemical evidence of the same phenomenon, as described in the preceding sections.

Microscopic examinations of rehydrated whole egg or yolk powder revealed only a few of the individual globules of oil found in the original fresh egg. It would seem apparent that the emulsion breakdown is not necessarily complete during drying, and that it continues during the subsequent storage of the egg powders, particularly at high temperatures.

This microscopic evidence offers further indication that the failure of dried whole egg powders to whip to a stable foam is centered around the presence of free oil, caused by the failure of the original emulsion to re-form upon rehydration.

Addition of emulsifying agents. The experiments in previous sections have demonstrated that the failure of dried whole eggs in foam tests, and consequently in certain baking tests, is probably related to the loss of emulsifying action and the liberation of free oil from the yolk. This free oil is absorbed by, and coats the surface of, the dry particles of whole egg powder and acts as an anti-foam agent when attempts are made to produce a stable foam as an aerating or leavening medium in sponge cake batters.

One approach to this problem would be to add emulsifying agents to take the place of the natural agent or agents, which are apparently partially destroyed in the drying operation or in the operations associated with drying of whole egg melange.

A 6-month-old sample of spray-dried whole egg powder was chosen for some of the initial investigations on the effect of added emulsifiers. This sample, when rehydrated and whipped, formed no foam. It is probable that the addition of sugar in the foam test would enable the production of some foam. However, the objective of these experiments was largely to ascertain the cause of poor foam, and sugar was not used. When 1% (rehydrated basis) of "Ultra Wet-K" (an alkyl aryl sulfonate) was added to the spray-dried whole egg powder during rehydration, the mixture whipped to a creamy, stiff foam with a foam specific volume of 4.2 cc. per g. This can be compared with a value of 8.7 cc. per g. for fresh egg, when whipped under identical conditions but without the additive.

This surface-active agent increased the apparent viscosity of the rehydrated egg and made the foam rather heavy. Therefore, more water was added to return the apparent viscosity to that more closely resembling the rehydrated egg without the sulfonate. With 20% more water, a foam with a specific volume of 5.90 cc. per g. was produced.

Similar results were obtained when "Nytron"[4] was used as an emulsifying agent, except that Nytron was required at levels of about 3% of the rehydrated whole egg melange. As with the Ultra Wet-K, the Nytron increased the apparent viscosity of the rehydrated melange of spray-dried eggs. Therefore, the addition of extra water aided in the production of a light foam. With 20% (rehydrated basis) extra water, the foam specific

[4] Nytron is the trade name of a surface-active agent. The active agents in this compound are sulfonated alkyl compounds. This product also contains sodium sulfate and sulfite.

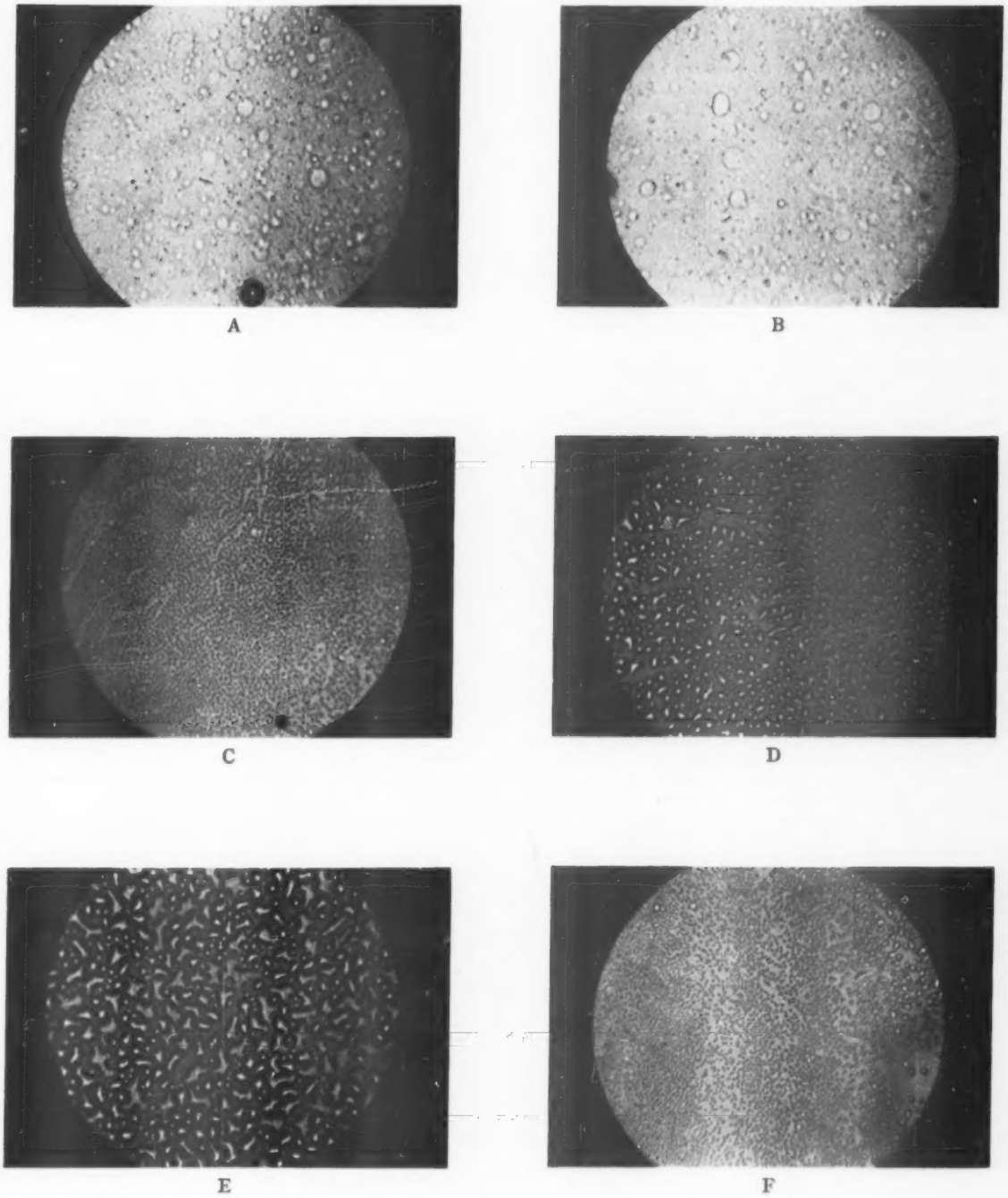

Figure 1. Photomicrographs of a film of egg yolk drying at room temperature on a microscope slide.

A, fresh egg yolk, showing spheroid oil globules in aqueous phase.

B, yolk as water starts to evaporate and oil globules distort to an ellipsoid shape.

C, when water no longer forms a continuous free phase, the oil appears as an exudation of droplets smaller than the original globules on the fresh yolk.

D, coalescence of droplets to larger, irregularly shaped globules.

E, oil collects as fairly large agglomerates.

F, several phases of drying, taken in one exposure. The degree of drying is at progressively advanced stages from left to right of photomicrograph.

volume was 6.5 cc. per g. With a total of 28% extra water, the foam specific volume was increased to 9.3 cc. per g. By adding increasing amounts of water to the point where the egg solids constituted 10% of the rehydrated mix instead of the usual 25%, the foam specific volume was increased to 18.5 cc. per g. Under these conditions, however, the foam was too weak for incorporation into a sponge cake batter.

Although these sulfonates served to illustrate the nature of the cause of poor foaming volume, they are not proposed as commercial additives to spray-dried whole egg powders. Therefore, other more generally admissible emulsifying agents were tried. These included monoglycerides, partial fatty acid esters of sorbitan, and polyethylene derivatives of these sorbitan esters.

The initial method of testing these emulsifiers consisted of adding them to a 6-month-old sample of spray-dried whole eggs, which had lost all power to whip to a foam unless sugar was added, but which could be readily whipped if the free oil was first removed with acetone. For example, about 0.5 g. of emulsifier was thoroughly mixed with 25 g. of dry whole egg powder in the bowl of an electric mixer. Water was then added (75 ml.) and the mixture whipped. No foam developed. More of the emulsifier was added in successive small portions, until a total of 4 g. had been added. In no case was there any development of foam on whipping the mixture.

In another experiment, the emulsifier was added to the liquid fresh egg (at a level of 1%) in a Waring blender, and the mixture was homogenized in a hand homogenizer. This mixture was dried and rehydrated for whipping tests. This treatment did not improve the foaming characteristics of the dry product.

A small amount of the homogenized liquid from the above preparation, before drying, was placed on a microscope slide and examined during drying, as previously described. This mixture did not show the characteristic fat exudation as was the case when no emulsifier was added. The experiment was repeated without the added emulsifier, that is, including the blending and homogenizing steps. In this latter case, without the emulsifier, there was a characteristic oil exudation during drying.

To elucidate further this apparently conflicting evidence, some of the sorbitan ester emulsifiers (for example, Tween and related compounds) were added to fresh, liquid eggs and the mixtures were whipped. No foam resulted. Two fresh eggs were whipped to a characteristic foam, and a small amount of Tween 80 (0.5% of the liquid) was added to the foam. The whipping was continued, and the foam gradually disappeared. In another experiment, the emulsifier was mixed with fresh, liquid egg, and the mixture was passed ten times through a hand homogenizer. This preparation, when whipped, also did not form any foam.

The above experiments indicate that the sorbitan fatty acid esters tried prevented oil exudation during drying and in that respect fulfilled their roles as emulsifying agents. However, those that were tried in this series of experiments acted as anti-foam agents in this particular application.

A sample of commercial soybean lecithin was used as an emulsifying agent. This additive did not materially aid the foam volume of the dried eggs when used at levels of from 0.5 to 1.5% of the rehydrated eggs. Qualitative experiments indicated similar results at higher levels.

A search for an edible emulsifying agent that is not an anti-foam agent is indicated, as the success of such a finding would, theoretically, be of great significance.

SUMMARY

Whole egg powders, even when freshly dried by lyophilization, showed an appreciable loss of quality in whipping characteristics.

Experiments in which egg yolk and egg white were dried separately and recombined with respective portions of undried egg indicated that the factor (or factors) affecting the loss of foam volume in whipping tests with rehydrated egg powders is associated with the yolk.

Dried whole egg powders, which had poor whipping characteristics when rehydrated, acted in themselves as anti-foam agents and caused the collapse of a preformed egg foam produced with fresh egg constituents. This anti-foam characteristic of these powders could be removed by treatment of the powders with fat solvents, such as acetone. Such solvent-washed powders whipped to voluminous foams characteristic of non-dried eggs.

The above experiments suggest that the free oil of dried whole egg powders is responsible for this anti-foam action. This was given corroboration by microscopic examination of a drying egg film, which showed a breakdown of the original fat emulsion and the formation of a free surface of oil.

Experiments with emulsifying agents indicated that certain types of emulsifiers can eliminate this anti-foam action of the free oil of egg powders. However, those emulsifiers that were found to be successful in this respect are questionable additives. Some of the emulsifiers are in themselves anti-foam agents and hence defeat their practical purpose. Further study of this latter phase of activities is indicated.

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Bactericidal and Drying Effects of Smoking on Bacon[a]

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Destruction of bacteria and loss in weight of bacon during experimental smoking were found to be correlated to some extent with smoke density, duration of smoking, temperature of the meat, and humidity of the smokehouse. Smoke density and temperature were the most important bactericidal factors. Weight loss was correlated primarily with humidity and was actually slightly retarded by the combined surface action of heavy smoke and high temperature.

For centuries, smoking has been used as a means of preserving protein foods. During the process food is subjected to the chemical constituents of smoke, to the elevated temperature of the smokehouse, and to a drying action. The modern tendency to rely on refrigeration as the principal method of preservation has reduced the importance of drying, and smoke is now used mainly to impart flavor to the product. It is generally assumed that the chemical constituents of smoke are largely responsible for its bactericidal action but few data are available to assess the part played by temperature and humidity. The present study is an attempt to evaluate the role of these factors in the destruction of bacteria during the smoking of bacon.

EXPERIMENTAL PROCEDURE

Plan of experiment. It was originally intended to carry out a factorial experiment involving the processing of duplicate pieces of meat, with and without smoke, at different temperatures and humidities. This proved impracticable because, even in the absence of smoke, odors and deposits could be detected on the meat from the tarry residues in the smokehouse. Attempts were made to avoid these effects by enclosing meat for "no smoke" treatments in heat-sealed Reynold's metal bags, but, despite circulation of clean air of house temperature and humidity through these bags, the internal humidity remained uncontrollably high. Comparisons were therefore restricted to 2 levels of smoking.

Temperatures and relative humidities attained in the smokehouse were: High temperature (131-142° F., 55-60° C.) and high humidity (50-100%); high temperature and low humidity (17-24%); and low temperature (68-103° F.; 20-40° C.) and high humidity.

For each experiment 2 or 3 pickled-cured pork bellies were cut into thirds and randomly allotted to the various predetermined combinations of smoke treatment and duration of exposure.

Measurement and control of smoke, temperature and humidity. The meat was smoked in a laboratory model smokehouse, 2 ft. 4 in. wide, 4 ft. long and 6 ft. high. During the smoking operation, air was continuously withdrawn from the bottom of the house, blown through thermostatically controlled heating coils, over wet and dry bulb temperature recorders, and then returned to the top of the house through a series of vents arranged to provide even distribution. A second wet bulb, adjacent to the recorder bulb, was connected to a solenoid valve which admitted sufficient steam to maintain the desired humidity. Since the air cooled slightly between the recorder bulbs and the house, the relative humidities reported were calculated from the humidity in the air duct and the temperature in the house.

Smoke was produced in a separate unit by burning mixed hardwood sawdust. Smoke density was controlled by means of a photoelectric cell mounted in the top of the smokehouse and connected through a General Electric smoke density recorder to a Modutrol valve, that allowed smoke to enter the air duct in front of the heating coils as required. The density of the smoke was measured in Ringleman units (0—no smoke to 5—complete blackness), and maintained at 0.5 to 0.8 unit during light smoking and at 2 units during heavy smoking. On the basis of the amount of phenol (approximately 4 γ per sq. cm.) deposited on the same kind of meat during commercial operations, the light smoking operation reported here (Table 2) is roughly comparable to commercial smoking although comparisons are difficult because of differences in scale, smoking time, etc.

House and meat temperatures were taken at 15-minute intervals throughout the smoking period. House temperature was measured with 3 thermocouples, one in the back top left corner, one at top center and the other in the front bottom left corner. Meat temperatures were measured with thermocouples inserted about ⅛ in. under the surface of lean portions of the meat.

Bacteriological and chemical determinations. Surface samples (3 sq. cm. in area and approximately 3 mm. thick) were removed from areas of lean with sterile steel cork-borers, scalpels and forceps. Five discs (15 sq. cm.) taken before smoking and 10 discs (30 sq. cm.) taken after smoking were used for bacterial counts. Samples were diluted in 2% salt solution and dispersed with the aid of coarse sand. Counts were made on duplicate plates after 5 days' incubation at 70° F. (21° C.) on agar containing proteose-peptone (0.5%), tryptone (0.5%) and salt (2%).

The relative amount of smoke deposited on the meat was assessed by determining the total phenols (6) in a water extract of 2 or 3 discs of meat. Because smoke constituents normally concentrate on the surface of smoked products (4, 5, 6), variations in the thickness of the discs were disregarded and results were expressed as γ per sq. cm. rather than per g.

RESULTS AND DISCUSSION

Temperature and humidity conditions during 12 experiments are given in Table 1. The house temperature increased steadily from room temperature (70-80° F.) and usually reached the desired level at the end of 3 hours' smoking. During the next 4 hours the house temperature increased slightly, generally not more than 5 degrees above the desired level, although higher temperatures were occasionally recorded for short periods. Since house humidity was often difficult to control, both the mean and the range observed during the final 4 hours of smoking are reported. Generally, meat temperature lagged below house temperature throughout the smoking period, but the lag was less marked at high humidities.

The relative amount of smoke deposited on the meat at 2 smoke densities is indicated by the phenol concentration on the surface (Table 2).

Bacterial survival. Bacterial survival was markedly affected by both smoke density and temperature (Table 2). The combined action of heavy smoke and high temperature reduced the number of bacteria 100,000-fold. In general, however, the survivals shown in this table are not directly comparable because of differences

[a] Issued as Paper No. 294 of the Canadian Committee on Food Preservation and as N. R. C. No. 3204.

TABLE 1

House conditions and temperature of meat during smoking

House conditions	Expt. No.	House temperature, ° F.		House humidity, during final 4 hrs. %		Meat temperature, ° F.	
		At end of 3 hr.	Maximum from 4 to 7 hrs.	Range	Mean	At end of 3 hr.	Maximum from 4 to 7 hrs.
Heavy smoke							
High temperature	A	132	140	17-18	17.8	100	116
Low humidity	B	134	139	19-33	26.0	98	120
High temperature	C	135	142	52-80	60.6	118	123
High humidity	D	134	137	86-97	89.6	129	131
Low temperature	E	76	80	74-86	78.6	71	75
High humidity	F	68	80	50-55	52.2	62	64
Light smoke							
High temperature	G	132	136	16-18	17.0	92	117
Low humidity	H	131	141	23-42	33.0	104	142
High temperature	J	134	138	88-93	90.8	131	134
High humidity	K	138	140	92-98	95.2	128	138
Low temperature	L	95	100	100	100	86	100
High humidity	M	103	103	99-100	99.8	90	100

in the temperatures and humidities actually attained in different runs. Statistical analyses of covariance (*3*, sec. 24-28) were therefore undertaken to assist in the interpretation of the data.

Log. percent bacterial survivals were found to be correlated significantly with all four of the factors—smoke density, duration of smoking, attained meat temperature and the absolute water vapour pressure deficit (wvpd). The analyses also yielded the following linear regression equations relating log. % bacterial survival (y) to attained meat temperature (x) in degrees F. and to the saturation deficit in water vapor pressure (w) measured in inches of mercury:

Light smoke,
3, 5 and 7 hours $y = 9.27 - 0.0826x - 0.025w$
Heavy smoke, 3 hours $y = 2.91 - 0.0351x - 0.188w$
Heavy smoke, 5 hours $y = 2.68 - 0.0351x - 0.188w$
Heavy smoke, 7 hours $y = 2.37 - 0.0351x - 0.188w$

As shown in Figure 1, light smoke caused little reduction in bacterial numbers at temperatures below 90° F. (32° C.), while heavy smoke caused significant reductions at the lowest temperatures studied (55° F.; 13° C.). However, at 140° F. (60° C.) either light or heavy smoke reduced the bacterial count to 0.01% of the original value. Bacterial survivals after light smoking were erratic and no significant differences could be attributed to duration of smoking. On the other hand, the destruction of bacteria by heavy smoke was proportional to the length of exposure, and survival after 5 and 7 hours' smoking was approximately 0.6 and 0.3 times that after 3 hours' exposure.

Bacterial survival was affected more by heat than by drying. The equations indicate that survival during light smoking was reduced on the average about 10,000-fold by a 50° F. elevation of meat temperature through the experimental range 90 to 140° F. (30-60° C.) at constant wvpd, but by less than 10-fold by an increase in wvpd of from 0 to 5 inches of mercury (the experimental range) at constant temperature.

Although the total number of bacteria was reduced by smoking, there appeared to be little selective action. Examination of several smoked and unsmoked samples

TABLE 2

Weight change, phenol content of surface and bacterial survival on bacon after exposure to heavy and to light smoke at various temperature and humidity conditions

House conditions	Time hr.	Heavy smoke				Light smoke			
		Expt. No.	Weight change, %	Bacterial survival, %	Phenol γ /sq. cm.	Expt. No.	Weight change, %	Bacterial survival, %	Phenol γ /sq. cm.
High temperature Low humidity	3	A	−6.8	0.02	....	G	−4.6	4.8	3
	5		−11.0	0.02	....		−7.5	1.8	2
	7		−10.4	0.003	74		−8.8	0.24	2
	3	B	−3.9	0.04	9	H	−2.3	.48	4
	5		−8.1	0.02	14		−6.1	.12	3
	7		−7.9	0.002	19		−6.0	.49	4
High temperature High humidity	3	C	−0.4	0.07	15	J	−1.4	0.010	5
	5		−2.9	0.02	28		−0.0	0.0010	5
	7		−4.7	0.008	30		−0.1	0.0002	4
	3	D	−0.3	0.04	12	K	+1.6	0.51	1
	5		−0.5	0.001	14		−1.2	0.11	2
	7		−0.7	0.003	14		−2.4	0.035	2
Low temperature High humidity	3	E	−0.7	7.1	....	L	+2.0	17.0	1
	5		−0.6	0.97	6		+1.7	61.0	2
	7		−1.3	0.62	12		+1.6	35.0	2
	3	F	−2.8	2.5	8	M	+1.6	85.0	1
	5		−3.1	6.1	12		+1.8	93.0	1
	7		−5.4	2.7	17		+2.3	71.0	1

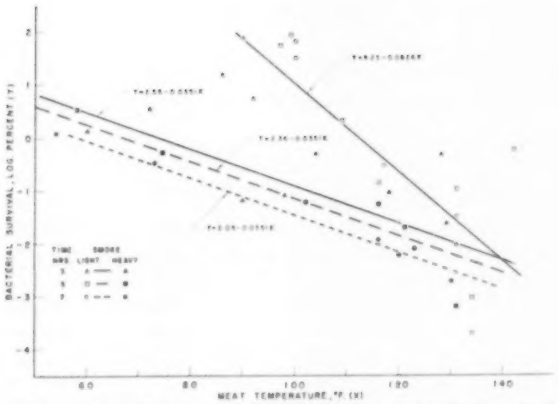

Figure 1. Effect of meat temperature on bacterial survival after light and heavy smoking, as shown by the regression of log. percent bacterial survival (y) adjusted to constant water vapor pressure deficit (w = 1.69) on meat temperature (x).

indicated that the proportion of rod forms and cocci was unaltered by smoking. However, pigmented cocci survived better than the non-pigmented species. Organisms which survived smoking were no more resistant to phenol (C_6H_5OH) than organisms isolated from unsmoked meat.

Although the concentration of smoke constituents on the surface gradually decreased due to diffusion (Table 3) and possibly oxidation, these constituents continued

TABLE 3

Effect of smoking bacon on moisture content and of smoking and storage on phenol content

	Surface 1/8"	Second 1/8"
Moisture (%)		
Initial [b]	74.08	68.63
Smoked 3 hr.	68.68	61.37
Smoked 6 hr.	66.94	66.06
Phenol (γ/sq. cm.)		
Smoked 3 hr.	10.8	0.2
Smoked 6 hr.	37.0	1.9
Smoked 3 hr. and stored		
1 week, 30° F.	4.7	3.3
3 weeks, 30° F.	3.3	2.8
5 weeks, 30° F.	2.4	3.2
Smoked 6 hr. and stored		
1 week, 30° F.	15.2	6.7
3 weeks, 30° F.	11.2	7.7
5 weeks, 30° F.	8.7	7.1

[b] Smoke density 2, house temperature 133-139° F., house humidity 18-26% RH.

to exert a bactericidal effect for some time after the meat was removed from the smokehouse. For example, an initial load of 7.3 million organisms per sq. cm. was reduced to 126,000 by smoking alone, to 49,000 after storage at 30° F. (—1.1° C.) for 22 hours and to 330 after storage for 4 days. On unsmoked bacon stored at 30° F. bacteria grow quite well *(1)*. To determine whether the continued bactericidal effect was due to smoke constituents alone, bacteria were sprayed onto a side, after removal from the smokehouse, increasing the load from 160 to 640 per sq. cm. The latter number was reduced to 40 after 22 hours' storage at 30° F. Similar residual effects of smoke have been noted by Jensen *(2)*.

Weight change. Smoking at high temperatures and low humidities resulted in an average loss in weight after 7 hrs. of 8.3% (Table 2). This was caused mainly by loss of water and partly by loss of fat and extractives. Accurate assessment of the depth of dehydration was difficult, but the greatest loss appeared to be from the 1/8-inch layer at the surface (Table 3).

Analyses of covariance indicated that changes in weight were also related to the 4 factors studied. The following regression equations relate the percentage change in weight (z) to the attained meat temperature (x) in degrees F. and the saturation deficit in water vapor pressure (w) in inches of mercury.

Light smoke,
3, 5 and 7 hours $z = 4.71 - 0.033x - 1.73w$
Heavy smoke, 3 hours $z = -3.75 + 0.050x - 1.96w$
Heavy smoke, 5 hours $z = -5.44 + 0.050x - 1.96w$
Heavy smoke, 7 hours $z = -6.34 + 0.050x - 1.96w$

From these equations it may be computed that increasing the wvpd from 0 to 5 in. mercury at constant temperature would increase the average loss in weight by 8 to 10%. It may be calculated also that increasing the attained meat temperature by 50° F. in the experimental range at constant wvpd would have increased the average weight loss by about 1.5% with light smoke, but would have reduced it with heavy smoke by about 2.5%. This differential effect may have been due to an increased surface glazing or tanning by the heavy smoke at the higher temperatures.

SUMMARY

Smoke constituents were chiefly responsible for the bactericidal effect obtained during the smoking of bacon. With dense smoke, high temperatures were more destructive than low temperatures, but variations in humidity had little effect. The bactericidal effects of light smoke were variable but the combination of high temperature and light smoke was almost as destructive as heavy smoke. However, at lower temperatures light smoke had little bactericidal effect.

Weight loss was inversely correlated with humidity in both heavily and lightly smoked samples. There was some indication that losses were greater during light smoking at high temperatures, but the loss did not appear to increase with time. The weight loss during heavy smoking increased with time of smoking but decreased with a rise in temperature, probably because the combination of smoke and high temperature tanned the surface and reduced the transfer of water.

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Antioxidant Effect of Liquid Smokes

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The antioxidant effects of four commercial liquid smokes are described. Antioxidant activity is shown to vary from zero to a pronounced effect. Smoke and ascorbic acid are shown to function synergistically. Artificial aqueous lard systems were employed, but the most promising of the commercial liquid smokes was tested in frozen ground pork.

Natural smoke is known to contain antioxidants which retard rancidity in meat, poultry, or fish subjected to the smoking process *(1, 4, 5, 9, 12, 18, 22, 23)*. These have not been identified, but since most of the known primary fat antioxidants are phenolic substances, it has generally been assumed that the constituents of smoke responsible for their antioxidant effect are phenols. Artificial smoke flavors or "liquid smokes," prepared by collecting at least some of the volatile products of the thermal decomposition of wood, have been on the market for more than half a century. A variety of commercial liquid smokes are now available for the flavoring of meat, fish, and other products. No information could be found on the effect of these liquid smokes on fat rancidity.

Although the chemistry of the thermal decomposition of wood has been studied by a number of investigators *(3, 6, 7, 8, 15)*, the subject is very complex; the smoke itself is variable in composition, and published information pertinent to the collection and modification of the smoke for use in flavoring and preserving foods is very limited. A brief resume of this literature as it applies to the preparation of liquid smokes follows.

When hardwood sawdust or chips are heated in a closed container the wood is converted into charcoal and a number of volatile constituents. With the exception of some gases, most of the latter can be condensed in a cooled receiver. The condensate (which presumably contains the smoke constituents which would be deposited on the exposed food during the natural smoking process) is composed of an aqueous phase known as pyroligneous acid and an immiscible tar. It is this condensate, usually in a modified form, which is the base of most commercial liquid smokes.

More than a hundred compounds have been identified in such condensates *(8)*. Some, notably the lower fatty acids, are detrimental to the odor *(7)*. Others, including methanol, acetaldehyde, formaldehyde, cyanderivatives, and creosotes, may be toxic and do not contribute to the desirable smoke odor. The fractions responsible for the smoke flavor may be concentrated by fractional distillation *(3, 6)* and extraction with selective solvents *(6)*. The concentrated oily material has an intense smoke odor and gives phenolic reactions. The phenol content of tissues subjected to the natural smoking process has also been found to correlate well with smoke odor *(14)*.

In addition to the possibilities for concentration and modification of the condensate, Pettet and Lane *(15)* have shown that the composition of the smoke itself may be materially altered by the conditions of combustion. Controlled air flow over the smoldering wood results in complete combustion of all the carbon to ash. In such smokes, the character of the smoke is changed by oxidation and condensation of some of the constituents. Phenols and formaldehyde condense to give resins which are probably responsible for the yellow color and gloss of smoked products. The odor of the condensate is milder and less acrid. However, a large number of the smoke constituents do not condense readily in the pyroligneous liquor. Pettet and Lane recovered these constituents by passing the smoke through a series of adsorbents, but their method would appear to be too complicated for other than research purposes. An apparatus has been described for collecting active smoke components by passing through an electric field causing deposition of smoke particles *(19)*.

All of the liquid smokes tested in this study are products of the thermal decomposition of hard wood, usually hickory. Only four commercial liquid smokes answering this description could be located at the time this work was initiated. All four were included in the study. Smoke flavored barbecue sauces, salts, etc. in which the smoke flavor was mixed with other condiments were not included.

The four liquid smokes available differed markedly in solubility, pH, phenol content, and in concentration and quality of the smoke odor as well as in the antioxidant properties of main interest in this investigation. In no case was it possible to obtain details of the manufacturing process which might throw light on these differences. Two of the manufacturers stated that they did not know the composition of their products. The other two failed to answer inquiries concerning composition. Although this lack of information makes it impossible to characterize and differentiate the various commercial products and discourages basic research on their properties, nevertheless it was believed to be worthwhile to determine whether the commercial liquid smokes now available have the antioxidant properties characteristic of the natural smoking process.

In addition to the determination of the direct effect of each smoke on rancidity, it was also considered desirable to investigate the antioxidant effect of combinations of the smoke with ascorbic acid. The latter has been proposed for use in meats to protect color, but its effect on pork fat has proved to be very erratic, sometimes accelerating and sometimes inhibiting rancidity. It is believed that these differences are due, at least in part, to variations in concentration of the natural phenolic inhibitor, tocopherol, in the meat fat. When tocopherol is low, ascorbic acid accelerates rancidity, whereas at higher tocopherol levels it acts as an antioxidant *(21)*. It was therefore considered possible that if phenolic antioxi-

TABLE 1

Characteristics of commercial liquid smokes

Code letter	Manufacturers' description of process used	Solubility	Approx. conc. for smoke flavor in meat	pH	Odor characteristics
A	Destructive distillation of hickory wood in a closed vessel. Condensate collected and liquid product made by use of emulsifying agent.	Soluble in water and in 20% NaCl. Slight precipitate from saturated brines on long standing.	0.1%	6.05	Rated lowest in smoke odor. Harsh. Described by various judges as suggestive of "tar," "creosote," "wet, burnt wood."
B	Highly concentrated. Contains no pyroligneous acid or formaldehyde.	Must be dissolved in small amount of alcohol or prop. glycol before diluting with water. Turbidity increases with salt concentration. Much precipitate with more than 10% NaCl.	.03%	4.9	Rated highest in desirable smoke odor. Mellow. Characteristic smoke odor.
C	Destructive distillation of hard wood.	Clear solution in water and in 10% NaCl. Progressively less soluble in more concentrated brines.	1.0%	3.3	Intermediate between A and B.
D	Extraction and concentration from hickory wood juice (pyroligneous acid). Methyl alcohol eliminated.	Same as C above.	1.0%	3.25	Intermediate between A and B. Similar to C.

dants are present in liquid smokes, they might act synergistically with ascorbic acid.

EXPERIMENTAL

Characteristics of commercial smokes. In view of the lack of information concerning method of manufacture or composition, the 4 liquid smokes available are designated throughout this study simply by code letters. Table 1 lists all of the information which could be obtained from the manufacturers as well as our own observations on some properties important in their selection and use.

The concentrations required to give a noticeable smoke odor varied widely in the different commercial preparations. The figures given are only rough approximations. Since the quality as well as the intensity of the odors varied, it was very difficult to match concentrations. Nine judges rated desirability of the smoke odors at the concentrations indicated. All agreed in rating A lowest, but there was less unanimity on the relative rating of the other 3 smokes. With the exception of B, all were soluble directly in water but showed increasing turbidity or separation in more concentrated salt brines. Variations in pH between 3 and 7 had no noticeable effect on solubility.

Antioxidant effect on lard in artificial systems. In order to obtain a rapid evaluation of the relative antioxidant effect of the various smokes, an accelerated test was performed using artificial aqueous fat systems. The method has been described in detail elsewhere *(13)*. The aqueous solution containing the compounds to be tested in 0.02 M phosphate buffers, pH 5.8, was absorbed onto a filter paper in a petri dish. Rendered pork fat containing .01% carotene as an oxidation indicator was melted and run onto a slightly smaller paper which was carefully placed over the first, eliminating all air bubbles. The petri dishes were sealed and stored in an incubator at 45° C. The melted fat very quickly distributes evenly over both papers in intimate contact with the aqueous solution. Peroxidation is accompanied by bleaching of the carotene, and the samples were designated rancid when the carotene was approximately half bleached as determined by visual inspection.

Each of the smoke flavors was tested alone, at a concentration within the range necessary to give a reasonably strong smoke flavor. In this and subsequent experiments, a 10% stock solution of smoke B was prepared using 5 parts propylene glycol, 4 parts water, and one part commercial flavor. This was further diluted with water and buffer to give the concentration shown. All others were dissolved directly in water. Each smoke was also tested in combination with ascorbic acid. The ascorbic acid was adjusted to pH 5.8 with sodium hydroxide before using.

Table *2* shows results of a typical experiment. The figures given are averages of duplicates which in every case differed by less than 20%. In several comparable experiments with other samples of pork fat, the same relative order of protection was found. With the exception of smoke A, which had no antioxidant effect either alone or with ascorbic acid, all smokes gave some protection, and smoke B a very strong protection. A similar strong protective effect was obtained with this smoke in subsequent experiments when the concentration was reduced to 0.01% of the aqueous phase, which was approximately the lower limit for a noticeable smoke flavor.

The synergistic effect of the liquid smokes with ascorbic acid is striking. The strong accelerating effect of ascorbic acid in aqueous solution in contact with fats low in phenolic inhibitors has been described in previous work *(13, 16, 21)*. All of the liquid smokes which by themselves have some antioxidant effect, and so may be presumed to contain phenolic inhibitors, give prolonged protection with ascorbic acid. The combination of ascorbic acid with smoke D was as effective as its combination with B in several experiments, although, when used without ascorbic acid, B consistently gave much greater protection than D.

TABLE 2

Antioxidant effect and phenol content of commercial liquid smokes

Liquid smoke added [a]	Days to turn rancid at 45° C.[b]		Phenols [d]
	No ascorbic	0.1% ascorbic	
Control, buffer pH 5.8 [c]	8	0.2	—
A. 0.1%	8	0.2	0
B. .04%	50	>90.0	+++
C. 1.0%	32	>90.0	+++
D. 1.0%	14	>90.0	+++

[a] Percentages represent final concentrations in aqueous phase.
[b] As indicated by half bleaching of carotene.
[c] All experiments carried out in .02M phosphate buffer, pH 5.8.
[d] Qualitative Millon's test.

The complete absence of antioxidant effect of smoke A, either alone or in combination with ascorbic acid, and the fact that it differed markedly from the other liquid smokes in odor characteristics, indicated that A might be lacking in phenolic constituents present in the others. A qualitative test for phenols was therefore run on each smoke as follows:

To 5 ml. of the diluted smoke (concentrations were the same as those used to test antioxidant activity, shown in Table *2*) was added 1 ml. of Millon's reagent (Eimer and Amend). Standard solutions containing .002 and .02% phenol respectively were treated in the same way. After mixing, all tubes were heated for a few minutes in a hot water bath (color comparisons must be made after relatively slight heating since precipitation of the colored complex occurs in the liquid smokes on more prolonged heating). Smokes B, C, and D all showed color development more intense than that obtained with the .02% phenol standard, whereas no color developed in smoke A either at this concentration or on repetition of the experiment with a 10-fold increase in smoke concentration.

Experiments on frozen ground pork. Since time did not permit an evaluation of all of the liquid smokes in meat, smoke B was selected on the basis of the foregoing trials on artificial systems. A 10% solution of liquid smoke was prepared as described above and further diluted with water or with ascorbic acid to the concentration desired before being mixed with the fresh ground pork. The total amount of liquid added was 2% of the weight of meat. An equal amount of water was added to the control and mixed for the same length of time. The meat was weighed into 50 gm. patties, packaged in polyethylene freezer bags with an outside waxed pasteboard container. These were frozen and held at —17° C. (0° F.).

In the first experiment on meat, the effect of smoke B at a concentration of .04% of the weight of meat was compared with that of butylated hydroxyanisol (BHA) and of ascorbic acid. The BHA was selected for comparison because it was previously found to be very effective in aqueous fat systems when added directly to the fat *(13)* and it has been widely used for the protection of animal fats *(10, 11)*. A 5% stock solution of the BHA in propylene glycol was prepared. However, upon further dilution with water the antioxidant separates out in oily globules. The uniform distribution of the BHA in ground meat is very difficult, and it is probably this factor which limits its effectiveness in meat.

The results, Table 3, demonstrated the prolonged protective effect of the liquid smoke. At the end of the 11 months' storage

TABLE 3
Comparison of several antioxidants on rancidity in frozen ground pork [a]

Antioxidant used	Peroxide number [c] after storage at —17° C. (0° F.)	
	6 months	11 months
Control	20.2	168
Ascorbic Acid, .1% [b]	61.2	223
BHA, .01%	7.9	72
Liquid Smoke B, .04%	0.0	0

[a] All samples contained 1% sodium chloride.
[b] Adjusted to pH 6.0.
[c] Milliequivalents per 1000 g.

period the smoked samples had an excellent fresh smoke odor and flavor whereas all other samples were extremely rancid. The accelerating effect of ascorbic acid alone was quite marked in this experiment. As previously pointed out, it may act either as an antioxidant or as a prooxidant in meat, depending upon characteristics (including the tocopherol level) of the meat itself.

It should be pointed out that oxidation of the fat in these experiments does not parallel oxidation of the hemoglobin to methemoglobin. Both BHA and also the liquid smoke accelerated pigment oxidation. At the end of 11 months the sample containing the smoke was completely brown, whereas the control was brown only on a thin external layer, retaining a pink core. From a practical standpoint this browning is probably more appropriate than the original fresh meat color when combined with the smoke flavor.

Table 4 records the results of a typical experiment on the synergistic effect of the liquid smoke with ascorbic acid in frozen ground pork. The level of liquid smoke was reduced to .02%

TABLE 4
Synergistic effect of liquid smoke and ascorbic acid in frozen ground pork [b]

Meat additives	Peroxide number [d] after storage at —17° C. (0° F.)	
	3 months	6 months
Control	12.2	38.1
Ascorbic Acid, 0.1% [c]	18.4	70.5
Liquid Smoke, B, .02%	6.0	20.0
Ascorbic Acid + Smoke [c]	0.0	5.9

[b] All samples contained 1% sodium chloride.
[c] Adjusted to pH 6.0.
[d] Milliequivalents per 1000 gm.

of the weight of meat in this experiment. This concentration gave a relatively mild smoke odor, and the protection obtained with the smoke alone was less than that previously obtained with the higher concentration. The ascorbic acid again caused acceleration, but the combination of smoke and ascorbic acid had a prolonged protective effect. A similar strong retarding action on rancidity was obtained in 3 other experiments in which ascorbic acid and the liquid smoke were combined in meat.

DISCUSSION

The foregoing experiments demonstrate that the effective antioxidant in the natural smoking process may be collected in the distillate from burning wood and concentrated. Furthermore, the antioxidant effect of the several commercial smoke flavors tested was roughly proportional to true smoke odor. The only liquid smoke tested which did not have an antioxidant effect was very different in odor characteristics from the others. It is probable that both antioxidants and constituents responsible for smoke odor lie in the phenolic fraction. A qualitative test for total phenols in commercial liquid smokes gave negative results for the smoke which lacked antioxidant properties, whereas the others were strongly positive.

Liquid smokes may not at present be used in meat products to be sold in interstate commerce. No doubt this is a wise restriction at the present time, since there is so little uniformity among commercial liquid smokes in qualty and intensity of the smoke odor and in antioxidant effect. The natural smoke is also known to inhibit bacterial spoilage *(9, 17)*. There is practically no published information on the bacteriostatic effect of liquid smokes although experiments are now in progress on this problem. The moisture loss which ordinarily accompanies the natural smoking process would not take place if liquid smokes were used. However, smoke constituents are much more important than the incidental drying in preservation, at least in fish *(17)*.

In spite of these uncertainties attending the substitution of the liquid smokes for the natural smoking process, there is much to be said in favor of their use both as flavoring and antioxidant. The natural smoking process deposits smoke constituents only on the outside of the treated meat. When hams or bacon are sliced, the cut surfaces are unprotected by smoke antioxidants. Much better distribution of the smoke might be expected if it were dissolved in the curing brine when pumping meats or allowed to penetrate with the salt in cover brines.

The amount (.01 to .04%) of the most concentrated of the commercial smoke flavors required to give an antioxidant effect is remarkably low and of the same order of magnitude as the pure phenolic inhibitors such as butylhydroxyanisol, nordihydroguariatic acid, gallates, etc. Unlike the gallates and other polyhydroxyphenols, the smoke constituents did not give purplish discolorations with iron salts. Furthermore, their much greater water solubility makes them infinitely easier to distribute in meat and fish. Further work on the structure of the antioxidant present in these liquid smokes would be of interest.

The synergism between the smoke constituents and ascorbic acid enhances the antioxidant effect of the

smoke and makes it possible to use ascorbic acid freely for color protection without danger of the frequently encountered accelerating effect of ascorbic acid on fat oxidation.

Possibilities for utilization of the antioxidant properties of the liquid smokes and of the synergistic combination of smoke and ascorbic acid are, of course, not limited to meat curing. Wherever rancidity is a problem and the smoke flavor desirable, their use is indicated. They have given excellent results (which will be reported separately) in protecting fat of frozen and salted fish. In addition to their use in raw meats or fish preserved in various ways, they may also be used in the cooking water of meats or seafoods which are to be preserved by freezing. Experiments are now underway on freezing preservation of artificially smoked shrimp.

SUMMARY

The antioxidant effect of four commercial liquid smokes was investigated in artificial aqueous lard systems. Their antioxidant activity varied from zero to a pronounced effect in concentrations as low as .01% of the commercial smoke preparation.

All of the smokes which exhibited antioxidant activity when used alone acted synergistically with ascorbic acid. The synergistic combination invariably had a strong antioxidant effect, although ascorbic acid alone accelerated rancidity.

The most promising of the commercial liquid smokes from the standpoint of antioxidant effect, concentration and desirability of smoke odor, was tested in frozen ground pork. As in artificial systems it had a very pronounced antioxidant effect on the meat. The combination of smoke and ascorbic acid functioned synergistically in meat as well as in artificial systems.

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Some Factors Affecting the Decomposition of Frying Fats

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Thermally induced changes in fats used for deep frying are described. Hydrolysis, polymerization, and oxidation take place simultaneously at elevated temperatures. A number of the objectionable developments—such as loss of rancidity resistance, color, and flavor changes—are shown to be associated with oxidative changes rather than hydrolysis. Means of restricting these changes are presented.

Almost universal acceptance of foods fried in deep fat has increased consumption to unprecedented heights during the past decade—and the trend continues upward. Improved frying techniques undoubtedly have been a dominant factor in the sudden and continued popularization of this convenient method of food cookery. Boggess and Ivy *(2)* in 1927 reported potatoes fried in both deep fat and shallow fat are more digestible than when prepared by boiling.

Commercial food preparation in restaurants, hotels, roadside stands, and in specialty houses featuring fried shrimp, clams, and chicken tend more and more to deep fat frying as a standard production method. The dough-

nut industry continues to grow at a rapid rate and potato chip consumption has increased year by year. New industries featuring frozen French fried potatoes, canned Chinese noodles, canned French fried onions, pre-fried canned and/or frozen meat balls and hamburger patties have expanded at a rapid rate since World War II. Appliances using deep fat as a means of heat conduction are now being used for "broiled" steaks and chicken and even for the frying of bacon.

Small sized, well designed, deep fat fryers are available in the household utilities section of nearly every department store. The availability of many foods—breaded and ready for the deep fat fryer—in the neighborhood grocer's frozen foods display case adds the factor of convenience to an already accelerating household trend.

This paper is concerned with problems arising from the thermal reactions of fats used for deep frying. Three general chemical reactions — all heat-induced — take place simultaneously when fats are used at elevated frying temperatures; namely, hydrolysis, polymerization, and oxidation. Much emphasis has been given to the hydrolysis reaction by past investigators probably because of the ease of measurement of free fatty acid development. It will be developed that hydrolysis of the frying fat represents but a minor portion of the total problem. It is indicated that oxidation of hot-frying fat is a much more important aspect of the problem than has been formerly recognized and that the problem of hydrolysis might be of secondary importance.

Arenson and Heyl *(1)* in 1943 reported important differences in the physical structure of doughnuts fried in various frying fats modified by addition of fatty acids and also by the presence of substances thermally developed in the normal process of frying. These authors concluded that the stability of shortening as measured by resistance to peroxide formation is without significance in the measurement of frying quality. These authors reported frying conditions in which the free fatty acid content did not develop to levels above 0.6%. No attempt was reported with reference to other decomposition products.

Fisher *(4)* in 1949 reported correlation between the thermally developed free fatty acids of frying fats and physical characteristics and fat absorption properties of doughnuts. Although the fats reported by Fisher did not develop free fatty acids above the level of 0.40%, he recognized as a possibility the influence of other thermally formed constituents in the frying fat.

Lantz and Carlin *(7)* in 1938 reported the rapid hydrolysis of fats when held at temperatures of 380° F. (193° C.) in the presence of a constant flow of steam. Hydrolysis was reported slow during the first 20 hours of the heating period but proceeded at a rapidly accelerating rate after free fatty acid levels of 0.5 to 1.0% were reached. This observation coincides with actual production experiences of manufacturers of fried foods from which large volumes of water must be evaporated. These authors, like Arenson and Heyl *(1)*, also reported that the more highly saturated hydrogenated fats developed free fatty acids at more rapid rates than did the less saturated fats.

Carlin and Lannerud *(3)* in 1941 reported rapid drop in the oxidative stability of fats subjected to deep fat frying temperatures and subsequent reduction in keeping quality of potato chips. Also noted were foaming tendencies of the frying fat, a decrease in iodine number, an increase in saponification number, and marked increase of refractive index.

Goodman and Block *(5)* in 1952 reported drastic changes in the shape, crust character, and fat absorption properties of doughnuts fried in fats subjected to prolonged heating at 375° F. (191° C.). Thermally formed substances in the fat were evidenced by increased free fatty acid content and greatly reduced iodine numbers.

Robinson, Black and Mitchell *(10)* in 1940 reported tendencies of various fats toward foam development when used for frying purposes after pre-heating periods at 375° F. (191° C.). Thermal decomposition was evidenced by increased refractive indices, lowered active oxygen values, and increased viscosity.

Numerous commercial producers of deep fat fried foods continuously encounter serious problems concerned with the stability of frying fats or rather with the lack of stability. Attempts have been made by the shortening manufacturers to solve these problems by means of improved manufacturing techniques. Although it is indicated that the rate of hydrolysis is not reduced by hydrogenation, Arenson and Heyl *(1)* and Lantz and Carlin *(7)*, the industry has nevertheless aimed toward improved hydrogenation selectivity with almost complete elimination of di- and tri-unsaturates from the glyceride mixtures. Many oil formulations have been studied including hydrogenated peanut, cottonseed and soyabean oils in pure form and in blends, together with lard, hydrogenated lard, and hydrogenated lard-vegetable oil blends. Finished products with active oxygen stabilities upwards of 200 hours are in common use despite failure to correlate active oxygen stability either with frying properties or foaming properties, Arenson and Heyl *(1)*, Carlin and Lannerud *(3)*, Robinson, Black and Mitchell *(10)*. Highly bleached, almost water white products are often manufactured especially for frying purposes, although there is no evidence that the absence of color pigments improves frying properties and it is recognized that dark colors are developed in the fat after several hours of use, Lantz and Carlin *(7)*, Carlin and Lannerud *(3)*, Robinson, Black and Mitchell *(10)*. The use of antioxidants in frying fats is increasing; however, it should be recognized that the antioxidant may be quickly lost at frying temperatures unless it is continuously replenished, Sair and Hall *(11)*, Magoffin and Bertz *(9)*, Kraybill, Beadle, Vibrans, *et al. (6)*.

SOME TYPICAL COMMERCIAL PROBLEMS

A few typical commercial problems brought to the attention of this laboratory may serve to indicate certain fundamental facts with respect to frying fats and their care.

PROBLEM:

Rapid Deterioration of Frying Fat in Household Type Deep Fat Cooker (2½-lb. fat capacity).

OBSERVATIONS:

1. Rapid development of free fatty acids.
2. Rapid increase of fat viscosity.
3. Rapid drop in smoke point.
4. Rapid increase in color of fat.
5. Foam development.
6. High wattage heating elements required to secure maximum speed.
7. Carbon encrustation of heating elements after three to four frying cycles.
8. Lack of fat replacement or turnover.

PROBLEM:

Rapid Deterioration and Foam Development in Frying Fat used for Processing French Fried Potatoes.

Kettle: Gas fired tubular design of 4000-lb. capacity.

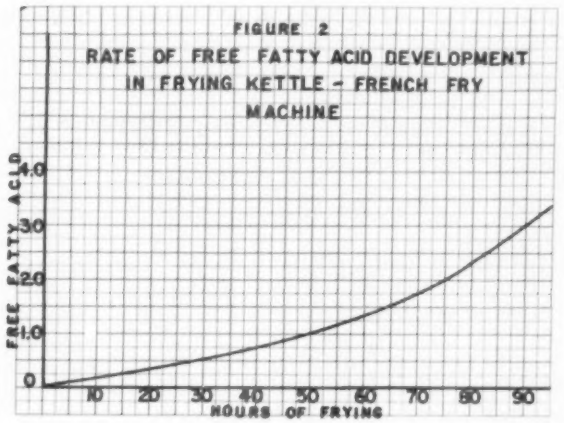
OBSERVATIONS (FIGURES 1 AND 2):

1. Fat turnover excellent; once in eighteen hours usage.
2. Severe encrustation and pitting of tubes of oil-fired heating mechanism.
3. Focal points of over-heating of tubes brought about through use of ceramic baffles.
4. Fat aeration during filtration.

FIGURE 1. FRYING FAT USED IN FRENCH FRY POTATO MACHINE
FRYING TEMPERATURE 360° F. TYPE OF FAT: HYDROGENATED COTTONSEED OIL

ANALYSIS	ORIGINAL FAT	4 HR USE	18 HR USE	48 HR USE	72 HR USE	85 HR USE	95 HR USE
FREE FATTY ACID	0.04	0.14	0.28	1.0	1.8	2.9	3.4
IODINE NO.	67.0	67.0	66.4	63.5	61.2	59.0	58.0
STABILITY (A.O.M)	22.6 HRS.	81	35	8	6	7	14
SAYBOLT VISCOSITY (150°F)	90 SEC	93	114	130	124	141	151
COPPER CONTENT	LESS THAN 0.5 P.P.M.						LESS THAN 0.5 P.P.M.
FOAMING TENDENCY	NONE	NONE	NONE	NONE	SL. FOAM	FOAM	STRONG FOAM
FLAVOR	GOOD	GOOD	GOOD	SL. ACRID	ACRID	ACRID	VERY ACRID

If the operation depicted above had been properly conducted, the turnover of frying fat (once in 18 hrs.) should have been adequate to maintain a free fatty acid equilibrium not higher than 1.0-1.5% for a much longer period of time than 48-72 hours. Examination of the frying kettle showed deep pitting of metal and a severe encrustation of carbon on the surface of the heating tubes immediately surrounding the gas burners. Surface temperature of the heating elements was found to be higher than 1200° F. (650° C.) as determined by thermocouple attached directly to tube surface. Lowered heat concentration and better heat distribution would have prolonged the usefulness of the frying fat.



Contrasted with the above, the installation of a heat exchange unit with a maximum temperature of 475° F. (246° C.) eliminated the foaming condition and trebled the period of usefulness of the fat.

PROBLEM:

Rapid Foam Development in Frying Fat.

Kettle Capacity: 6900 lbs.

Fat Turnover: Twice in 10 hours frying time

Type of Fats: Hydrogenated peanut oil
Hydrogenated cotton oil

OBSERVATIONS (FIGURES 3, 4, 5, 6, 7):

This problem was associated with early development of foam and objectionable flavors in fat used in frying a farinaceous product of relatively low moisture content. Despite an unusually high rate of fat turnover, despite low temperatures in the heat exchange unit (450-475° F.; 232-246° C.), severe foam developed in the frying fat after 24-36 hours use. More than 50,000 lbs. of frying fat was used in this test.

It was evident from viscosity changes and drop in iodine number that an unusual degree of oxidation and polymerization was probably responsible for both foam and flavor problems. Examination of the frying equipment led to discovery of a cascade effect occurring during the circulation of the hot frying fat through the system. The fat was being continuously aerated at frying temperatures of 360° F. (182° C.).

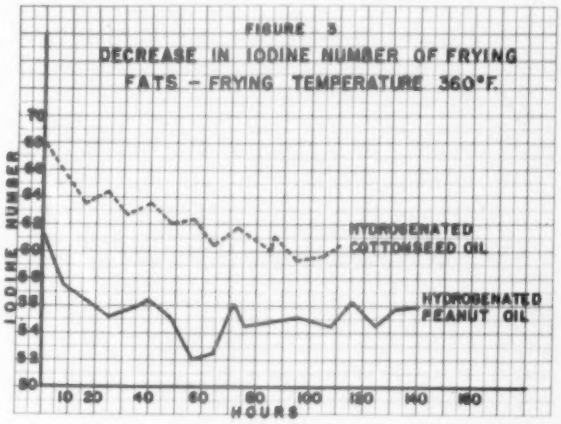


FIGURE 3
DECREASE IN IODINE NUMBER OF FRYING FATS - FRYING TEMPERATURE 360°F.
IODINE NUMBER
HYDROGENATED COTTONSEED OIL
HYDROGENATED PEANUT OIL
10 20 40 60 80 100 120 140 160
HOURS

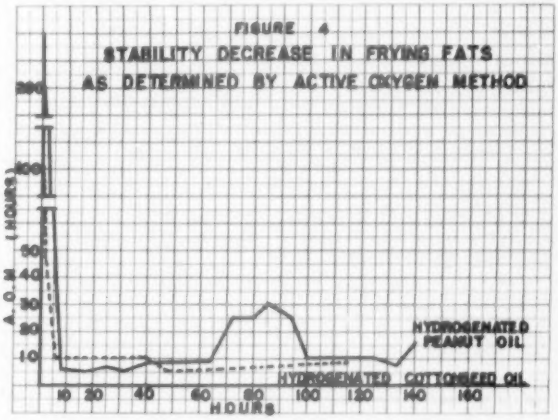
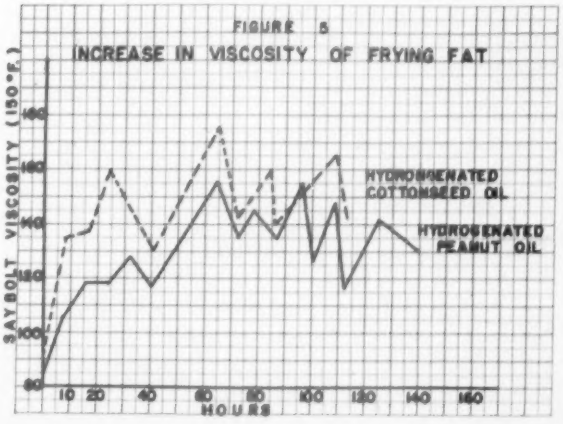
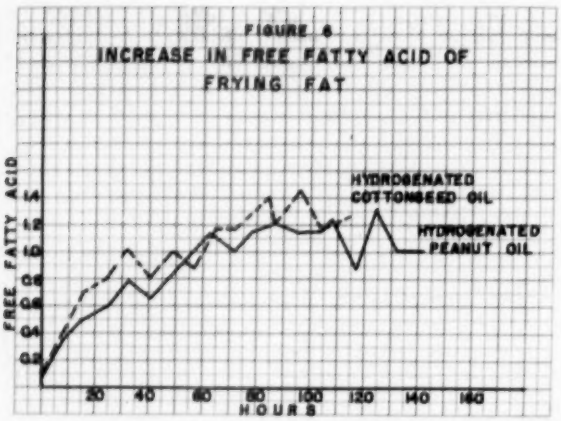
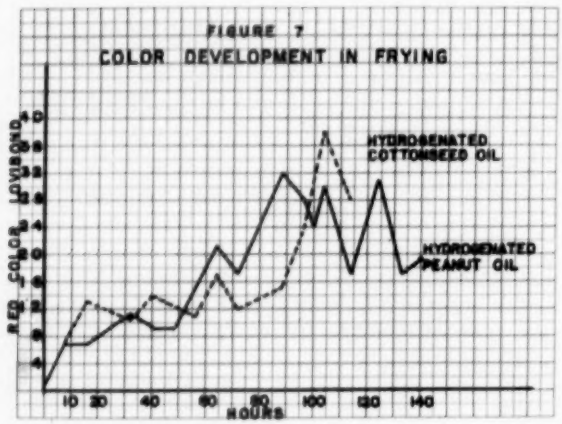


FIGURE 4
STABILITY DECREASE IN FRYING FATS AS DETERMINED BY ACTIVE OXYGEN METHOD
A.O.M. (HOURS)
HYDROGENATED PEANUT OIL
HYDROGENATED COTTONSEED OIL
10 20 40 60 80 100 120 140 160
HOURS







Consideration of such examples of improperly planned frying operations leads one to several questions with respect to the thermal reactions of frying fats.

1. What is the influence of air on the stability of fats held at frying temperatures?
2. Is decomposition, caused by excessive heat and by oxidation, of greater importance with respect to frying fat stability than hydrolytic action due to the presence of moisture?
3. What substances are responsible for the foaming tendencies of heated fats?
4. Are these substances responsible for changes in frying properties of fats? Do they influence the crust structure of doughnuts and potato chips?
5. Would removal of the air-fat interface result in increased stability in frying fat?

Tests were arranged to study several of these questions. Two Hotpoint (electrically heated) deep fat fryers of 25-lb. fat capacity were regulated thermostatically at 360° F. ± 5° (182° C. ± 2.8°). Hydrogenated cottonseed oil was used as the frying fat. Four-ounce quantities of potato chips were fried in the fats twice daily. Fat turnover was negligible. The fat of one kettle was protected from exposure to air by means of a metal float or "boat" constructed to cover the surface of the frying fat to within ¼ inch of the edge of the kettle. The fat in the second kettle was permitted to stand unprotected from the atmosphere during the entire heating interval. Samples were taken periodically for a period of 9 days and were subjected to chemical analysis (Figures 8 and 9).

Figure 10 indicates that the keeping quality of fried foods should be greatly increased if the frying fat could be protected from atmospheric oxygen. It is interesting to observe that the keeping quality of chips fried in highly oxidized fats is greater than the keeping quality of chips fried in less highly oxidized fats. This observation is in agreement with data from several preceding tests.

Further tests were made using fats contaminated by addition of oxidized drippings taken from the exhaust flue of a commercial potato chip frying machine. It was found that the presence of 0.75% of exhaust drippings (3 oz. per 25 lb. fat) greatly accelerated the rate of foam development, rate of color development and to a smaller degree the rate of free fatty acid formation. Exhaust contamination is not the only phase of this problem. In commercial operations, attempts are made to "feed back" old or spent fats after the machine has been loaded with fresh fat.

Oxidized fatty acids were extracted from the heated fats reported in Figures 8 and 9 and also from the fat contaminated with 0.75% exhaust drippings. Extraction was made according to the method of Leukowitsch in 1921 *(8)*.

FIGURE 8. CHEMICAL CHARACTERISTICS OF FRYING FAT HELD AT 360° F. (FAT SURFACE UNPROTECTED)

FAT	FREE FATTY ACID	STABILITY (A.O.M.)	COLOR (RED ONLY)	IODINE NO.	SAYBOLT VISCOSITY (210°F.)	SAP. NO.	FOAMING PROPERTIES
ORIGINAL HYDROGENATED COTTONSEED OIL	0.04%	70 HRS.	1.8	75	52	191.3	NONE
24 HRS.	0.14%	25 HRS.	5.7	72	65	-	NONE
48 HRS.	0.26%	10 HRS.	9.1	70.8	74	-	SL. FOAMING
96 HRS.	1.2 %	27 HRS.	TOO DARK	68	-	-	FOAMING
120 HRS.	1.2 %	36 HRS.	TOO DARK	62.1	-	-	INTENSE
144 HRS.	1.8 %	120 HRS.	TOO DARK	55.4	180	-	INTENSE
216 HRS.	2.3 %	165 HRS.	TOO DARK	54.0	272	207.9	INTENSE

FIGURE 9. CHEMICAL CHARACTERISTICS OF FRYING FAT HELD AT 360° F. (FAT SURFACE PROTECTED BY FLOAT)

FAT	FREE FATTY ACID	STABILITY (A.O.M.)	COLOR (RED ONLY)	IODINE NO.	SAYBOLT VISCOSITY (210°F.)	SAP. NO.	FOAMING PROPERTIES
ORIGINAL HYDROGENATED COTTONSEED OIL	0.04%	70 HRS.	1.8	75	52	191.3	NONE
24 HRS.	0.07%	23 HRS.	4.3	-	-	-	NONE
48 HRS.	0.13%	38 HRS.	6.0	-	-	-	NONE
96 HRS.	0.83%	25 HRS.	9.3	-	-	-	NONE
120 HRS.	1.40%	17 HRS.	4.7 X	-	-	-	NONE
144 HRS.	1.5 %	17 HRS.	6.8 X	75.1	54	-	NONE
216 HRS.	2.0 %	19 HRS.	9.1 X	74.5	52	194.6	NONE

X — 1 INCH COLUMN

FIGURE 10. POTATO CHIPS INCUBATED AT 145° F. IN NEW GLASS JARS (2200 ML. CAPACITY) 10 G. CHIPS PER JAR. RANCIDITY DETERMINED BY ODOR.

CHIPS-FRIED AFTER HEATING FAT AT 360° F	WITH PROTECTIVE FLOAT	WITHOUT FLOAT
24 HRS. HEATING	57 DAYS	21 DAYS
48 HRS. "	56 DAYS	* 4 DAYS
72 HRS. "	58 DAYS	* 4 DAYS
96 HRS. "	54 DAYS	* 16 DAYS

*** Crust structure of chips extremely porous. Crust color dark brown.**

Results of this analysis are given in Figure 11.

Only very small quantities of oxidized fatty acids were developed in the fat when the air-fat interface was reduced by means of a protective float. Where the air-fat interface was not reduced oxidation products were formed in appreciable

FIGURE 11. OXIDIZED FATTY ACIDS OBTAINED FROM FRYING FAT HELD AT 360° F.

SAMPLE	OXIDIZED FATTY ACIDS	SAP NO
1. CONTROL SHORTENING HYDROGENATED COTTONSEED OIL	-	191.3
2. SAME AT 128 HRS. (EXPOSED TO AIR)	7.8	200.1
3. SAME AT 128 HRS. (PROTECTIVE LID)	0.14	193.0
4. SAME AT 128 HRS. PLUS 0.75% EXHAUST DRIPPINGS (EXPOSED TO AIR)	9.7	205.7
5. OXIDIZED FATTY ACIDS FROM SAMPLE NO. 2	-	202.8

amounts. When contaminated fat and exhaust drippings were added to the fat the rate of formation of oxidation products was increased. These changes were confirmed organoleptically by changes in the flavor and appearance of the fat. Surface appearance of potato chips, doughnuts and other foods fried in the highly oxidized fats confirmed the findings of Goodman and Block *(5)*.

SUMMARY

Changes resulting in foam development, changes in physical properties of the fried food, loss of rancidity resistance in both fat and fried food, color development and objectionable flavor development are associated with thermally induced oxidative changes rather than hydrolysis. These thermally induced changes may be restricted to practical limits if the following conditions are maintained:

1. Rapid rate of fat turnover.
2. No prolonged heating of fat without compensating turnover.
3. Non-aeration of hot fat during circulation through heat exchangers, filters, the frying equipment, or by the "cascading" of hot fat into holding tanks.
4. Avoidance of intense heat input in the attempt to secure additional frying capacity.
5. Removal of improperly designed heating elements which result in intensely heated focal points.
6. No contamination of fat through drip back from exhaust vents.
7. No contamination of fresh fat through "feed back" of old or spent fat.
8. Control of chemical contamination from products fried in the fat.
9. Control of deposited food fragments during frying.
10. No contamination with cleaning and filtering compounds.

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Canning of Acidified Banana Puree

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(Manuscript received July 13, 1953)

A canning procedure for packing acidified banana puree is described. Means of overcoming or minimizing the usual problems encountered—pink discoloration, for example—are suggested. Blanching, acidifying, addition of sugar, and processing are discussed.

During the past several years interest in the canning of bananas, especially in a pureed form for baby food, has increased. Banana puree is not a new product; numerous attempts have been made to can a puree suitable for the ice cream and bakery trade. For the most part these attempts were only moderately successful. A main reason why canning has not received more attention is that under normal economic conditions fresh fruit is available the year round. In addition, since there are no commercial plantings in the United States, the industry—even if developed—would be entirely dependent upon importations.

Much information on the structure and development of the fruit, its chemical composition, and changes during ripening is available. This information has been

* Now located with the Burton Belt Company, Chicago.

collected and presented by von Loesecke *(4)*. The following information on the chemical composition of the banana fruit will provide a background for presenting the problems involved in canning banana puree.

The moisture content has been determined for a number of varieties of bananas. Stratton and von Loesecke *(3)* reported that the moisture content will range from 60 to 80% depending upon the variety. They also reported that the moisture content of the pulp increased as the fruit ripened. The most conspicuous change which occurs during the ripening process is the conversion of starch to sugar. Poland *et al. (2)* found that the total sugar content of the variety Gros Michel increased from approximately 2% at the time of unloading to approximately 20% ten days later.

In most varieties the acidity of the fruit increases to a peak and then decreases slightly as the fruit ripens. Stratton and von Loesecke *(3)* state that in general the rate of acid production, as well as its later decrease, follows the same trend as starch hydrolysis; that is, when the hydrolysis is proceeding most rapidly, the acidity increases most markedly, and as the rate of hydrolysis is reduced, acidity becomes progressively less.

Several types of product discoloration have been encountered in the production of banana puree. A brown discoloration, attributed to a very active oxidative enzyme system, can be eliminated by inactivating the enzyme either with a hot water or steam blanch before pulping. A pink discoloration has also been noted. In 1947 the Research Division of the Continental Can Company, Inc. *(1)* showed that the presence of peel rag and either tin or zinc in the puree caused this type of discoloration. It was noted that the discoloration could be held to a minimum if the temperature of the banana was rapidly raised to 185° F. or above during blanching and maintained at that temperature until the final cooling of the canned product.

Because of the increased interest in a canned banana product, and as a continuation of earlier studies, an investigation was undertaken to study and to overcome the problems involved in packing a good quality banana puree. As a result of this investigation the following canning procedure was developed for packing acidified banana puree:

CANNING PROCEDURE

Good mature raw stock was selected. (Color index Number 6 and 7. "Banana Ripening Manual" Circular No. 14, page 6. The Fruit Dispatch Company, N. Y.). The bananas were carefully peeled with attention to removal of peel rag and rot. The bananas were blanched whole in steam or boiling water (or a combination of both) until a center temperature of 190° F. was reached. A medium size peeled banana requires about 6 to 8 minutes to attain this center temperature. The blanched bananas were checked for sugar content and pH value. They were pureed or macerated to the desired viscosity in a comminuting machine. Either during the comminuting process or after it the computed amounts of sugar and citric acid were metered and blended into the banana puree to obtain the desired sugar level and the necessary pH of 4.1 to 4.3. The puree was heated to approximately 200° F. and filled into cans. The cans were closed immediately, inverted and held for 5 minutes followed by water cooling to 100° F. The entire operation from peeling to filling into cans was carried out as rapidly as possible to insure a high quality product.

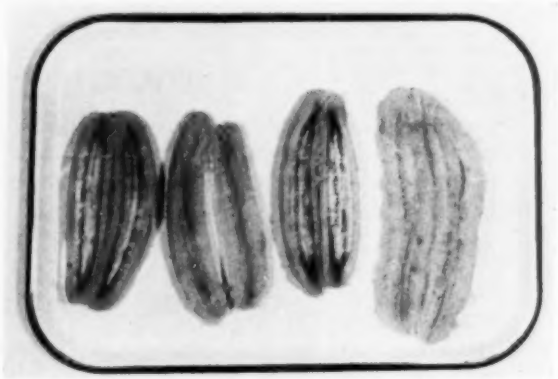
Blanching. As has been previously indicated, one of the problems encountered in canning banana puree is discoloration. For the most part this can be eliminated by blanching the peeled bananas in either steam or boiling water before they are pulped and exposed to air. Table 1 and Figure 1 show the effect of blanch time on the center temperatures of ripe bananas to be used for puree. Bananas which were peeled and heated to a center temperature of 180° F. or less showed a marked tendency to discolor after exposure to air for less than five minutes. Bananas which were peeled and heated to a center temperature of 190° F. did not discolor after exposure to air for periods of up to one hour.

TABLE 1

Effect of blanch time on color of ripe bananas to be used for puree

Time (minutes)	Peeled		Unpeeled	
	Center temperature	Color	Center temperature	Color
Boiling water blanch				
5	170° F.	Poor	.....	.....
6	184° F.	Good	160° F.	Poor
7	186° F.	Good	170° F.	Poor
8	190° F.	Good	177° F.	Poor
9	.....	.....	180° F.	Good
10	.....	.....	190° F.	Good
Steam blanch				
2	120° F.	Poor	115° F.	Poor
3	130° F.	Poor	120° F.	Poor
4	155° F.	Poor	140° F.	Poor
5	195° F.	Good	145° F.	Poor
6	198° F.	Good	170° F.	Poor
7	200° F.	Good	175° F.	Poor
8	202° F.	Good	190° F.	Good

Both steam and hot water blanching as well as blanching the bananas unpeeled and peeled were investigated. Obviously, the length of blanching time necessary to reach a desired temperature was greater for the unpeeled than for the peeled bananas. In addition it was noted that the bananas which were blanched unpeeled were more difficult to peel and to handle after blanching. Little difference was noted between steam and hot water blanching. In both methods it took an average of between 6 and 8 minutes to reach the desired center temperature of 190° F.



1 2 3 4

Figure 1. Effect of blanching on brown discoloration of bananas.

Legend:

(1) and (3)—Steamed blanched for four minutes.

(2)—Steam blanched for seven minutes.

(4)—Water blanched for seven minutes.

The blanching time should be controlled by the rate of heat penetration to reach a center temperature of 190° F.

Acidifying. In order to eliminate the need for a lengthy processing time which would adversely affect the quality of the final product, the pH of the puree was lowered to an optimum level of 4.2 by the addition of citric acid. By increasing the amount of acid in the puree to a point where the pH is below 4.5 the puree can be filled into the cans hot (200-210° F.), inverted, held for several minutes and then cooled. This type of process is based upon the observation that most bacterial vegetative cells are easily destroyed at temperatures of 200° F. if the pH of the medium is below 4.5. The growth of viable spores normally associated with canned food spoilage is also inhibited in a medium where the pH is below 4.5.

The amount of citric acid needed to lower the pH to the optimum level of 4.2 was computed for 100-pound batches of bananas from the curve in Figure 2. This curve was obtained

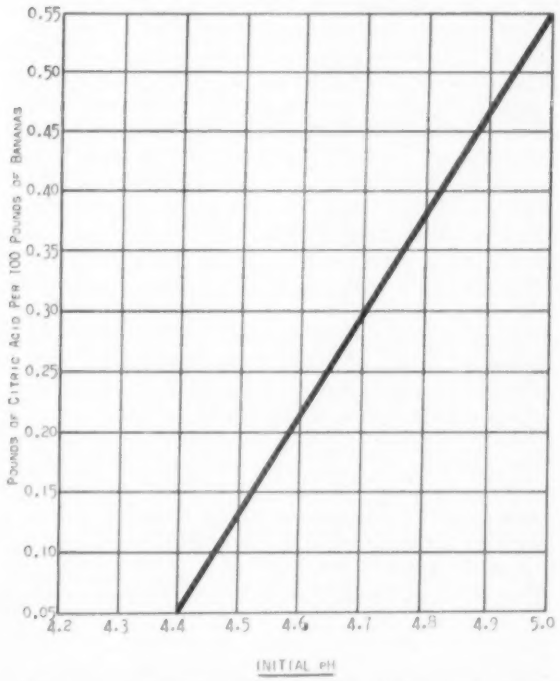


Figure 2. Nomograph for citric acid addition to lower pH to 4.2 for acidified banana puree.

from the pH levels reached by adding known amounts of citric acid to blanched bananas. Five varieties were studied: Fortuna, Cavendish, Calfiti, Santa Marta, and Golden Beauty. Although the figure shows a straight line function, the data reveal a slightly curved function; however, in the pH range which is most commonly experienced—i.e., pH 5.0 to 4.2—the curve can be considered a straight line function. Results of test packs have shown that the pH will rise as much as 0.5 as a result of the natural buffering action of bananas. Therefore, it is suggested that a minimum of 0.05 pounds of citric acid per 100 pounds of bananas should be added to compensate for the rise of pH during the canning process.

Therefore, to pack acidified banana puree the pH was lowered to 4.2 by the following method.

a. The pH was checked for each batch of bananas to be used for banana puree.

b. The amount of citric acid to be added was determined by referring to Figure 2.

Sugar addition. To make the product more pleasing from a flavor standpoint sugar may be added. The amount to be added is dependent upon the initial sugar content of bananas and upon the final concentration desired. Experimental packs have shown that a final product containing approximately 30 to 35% sugar will mask the slight citric acid flavor obtained from acidification.

The amount of sugar to be added to a desired level can be determined by referring to Table 2. For the experimental packs

TABLE 2

Sugar addition chart for acidified banana puree

Initial sugar content percent	Sugar content desired/100 pounds of blanched bananas		
	30% Pounds of sugar to add	35% Pounds of sugar to add	40% Pounds of sugar to add
5	25	30	35
7	23	28	33
9	21	26	31
10	20	25	30
11	19	24	29
12	18	23	28
13	17	22	27
14	16	21	26
15	15	20	25
16	14	19	24
17	13	18	23
18	12	17	22
19	11	16	21
20	10	15	20
21	9	14	19
22	8	13	18
23	7	12	17
24	6	11	16
25	5	10	15

the sugar and acid were mixed together as a thick paste to insure proper blending. Water in the amount of 1/5 to 1/8 of the calculated weight of sugar was added to the sugar and acid to make the paste. More water or liquid sugar could be added if a lower viscosity was desired. The sugar-acid paste can be added either before or after pulping but should be thoroughly blended with the pulped bananas.

Processing. Following pulping the puree was heated to 210° F. then filled hot into cans, closed, inverted and held for 5 minutes to sterilize the cover, then water cooled to a center temperature of 100° F. It might be necessary to give small size cans a short boiling water process since the covers might not be sterilized by inverting and holding.

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Colorimetric Method for Detection of Microbiological Spoilage in Citrus Juices[a,b]

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Off-flavors in citrus juices and concentrates may result from the growth of acid-tolerant bacteria, such as Lactobacillus or Leuconostoc. A rapid colorimetric method has been developed for the detection of this type of spoilage in citrus products, prior to, during, or after processing. The method, an application of the Voges-Proskauer reaction, is based on the detection of acetylmethylcarbinol or diacetyl, these substances being end products of bacterial metabolism. Comparison is made of the colorimetric method with other tests for detecting spoilage in citrus products.

Hays and Riester *(4)* and Murdock, Troy, and Folinazzo *(5)* have shown that off-flavors develop in orange concentrate because of the growth of certain acid-tolerant bacteria of the genera *Lactobacillus* and *Leuconostoc*. Growth of microorganisms of these genera in commercial evaporators during the first stages of concentration can result in spoilage of product on account of off-flavor development. Various factors, such as total bacterial population, bacterial strains, pH of juice, evaporator design, and length of processing time in the evaporator, may affect the rate of bacterial growth and hence the development of objectionable flavors; therefore, occurrence of spoilage in citrus products during commercial concentrating operations is erratic and unpredictable.

At present, microscopic examinations (direct counts) on samples of concentrate from evaporators are being made in some commercial plants to detect presence of bacteria that may cause spoilage. Proper interpretation of such microscopic examinations requires considerable experience on the part of laboratory technicians. Off-flavor development can eventually be detected by tasting samples of concentrate from the evaporator, but when detected by this method, spoilage frequently has occurred to such an extent that the product must be discarded. Also the reliability of an individual's opinion of the flavor of a product is subject to many factors that vary from day to day. Therefore, there has been no completely satisfactory method that commercial processors could readily adopt as a means for detection of growth of those microorganisms that may cause spoilage of citrus concentrate during processing.

This report discusses results of an investigation begun in April, 1952, to obtain a rapid and reliable chemical method for detecting the growth of microorganisms capable of causing off-flavors similar to buttermilk in orange concentrate. Such a method has been developed. It depends on the production by microbial activity of substances that can be detected easily and accurately by a colorimetric method.

One of the products of the metabolism of certain types of bacteria is acetylmethylcarbinol ($CH_3CHOH\text{-}COCH_3$). To be specific, *Lactobacillus plantarum*, one of the bacteria isolated and described by Hays and Riester *(4)*, when grown in single-strength juice or 20° Brix orange concentrate, produces this chemical compound when it utilizes substances present in the juice. The acetylmethylcarbinol can be changed into either diacetyl ($CH_3COCOCH_3$) or 2, 3-butylene glycol ($CH_3CHOHCHOHCH_3$) by further bacterial action or auto-oxidation. Diacetyl is one of the substances produced in butter starters and one of the principal compounds which gives butter its flavor. It is very volatile and is detectable by its characteristic odor when present in a product in very low concentrations. Although desirable in butter, it is quite undesirable in orange juice. If diacetyl is produced in orange concentrate during processing, the aroma and flavor imparted to the product is quite similar to that of buttermilk.

Acetylmethylcarbinol can be detected chemically by oxidizing it to diacetyl in the presence of atmospheric oxygen and strong alkali. The diacetyl may then be quantitatively determined colorimetrically *(7)*, since it forms a red compound in the presence of alkali, creatine and alpha-naphthol. This method is based on modifications of the original observation of Voges-Proskauer *(6)* for differentiation of *Aerobacter aerogenes* and *Escherichia coli*. This chemical method for either acetylmethylcarbinol or diacetyl has been applied for detection of these substances in citrus juices as an indication of the growth of bacteria that produce "buttermilk" off-flavors.

EXPERIMENTAL PROCEDURE

Chemical method. *Basis for Test.* Diacetyl is formed when a solution of acetylmethylcarbinol is treated with strong alkali in the presence of air. In the presence of alkali, creatine and alpha-naphthol, diacetyl forms a compound that results in a red solution, the intensity of which may be measured colorimetrically. Two procedures are given, since both were used in this investigation. Procedure A was initially developed and used for obtaining data presented in Tables 1 and 2. Procedure B is an adaptation and modification of Procedure A that was first used by Mr. Ellis M. Byer, Florence Foods, Inc., Florence Villa, Florida, as a control procedure during production of commercial orange concentrate. Procedure B is definitely more sensitive than Procedure A as is shown by the data in Table 3.

Reagents. Alpha-naphthol solution. Dissolve 5 g. of alpha-naphthol (Eastman 170) in 100 ml. of 95% ethyl alcohol.

Concentrated alkali solution containing creatine. Dissolve 40 g. of KOH in sufficient water to make 100 ml. of solution and then add 0.3 g. of creatine.

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[b] Cooperative research project of Florida Citrus Commission and Florida Citrus Experiment Station, Lake Alfred, Florida. Florida Agricultural Experiment Station Journal Series, No. 169.

[c] Florida Citrus Commission.

[d] Florida Citrus Experiment Station.

Powdered pumice stone.

The reagents should be prepared daily or may be kept for two weeks if refrigerated.

Procedure A. Reconstitute sample of citrus concentrate to be tested to 12° Brix ± 0.2°.

Place 5.0 g. of pumice and 20.0 ml. of reconstituted juice in a 25 mm. x 200 mm. test tube.

TABLE 1

Off-flavor development, diacetyl content, and microbiological counts in pasteurized 20° Brix orange concentrate, pH 3.9, after inoculation with Lactobacillus plantarum and processing in evaporator [a]

Processing time	Off-flavor development	Diacetyl content Procedure A	Direct microscopic count	Plate count
hr.		p.p.m.	Average of 25 fields	Orange serum agar, pH 5.4
Run No. 1				
Initial	None	0.6	0.3	102,000
5	None	0.6	0.4	.....
6	None	0.0	1.2	.....
7	None	0.0	1.3	840,000
8	None	1.6	1.4	1,205,000
9	None	1.0	2.6	.....
10	Slight	1.5	3.2	3,060,000
11	Slight	3.2	4.8	.....
12	Slight	3.4	7.1	.....
13	Slight	6.2	9.2	7,500,000
14	Definite	8.1	14.5	.....
Run No. 2				
Initial	None	0.4	0.4	725,000
1	None	0.2	1.3	.....
2	None	0.7	2.9	.....
3	None	1.7	6.0	.....
4	Slight	7.6	13.5	13,800,000
5	Definite	17.0	26.2	35,600,000
6	Definite	40.2	40.4	86,000,000
Run No. 3				
Initial	None	None	0.2	457,000
1	None	None	.....	.....
2	None	None	.....	.....
3	None	2.7	.....	.....
4	Slight	13.1	16.2	23,800,000
5	Definite	28.7	15.3	10,500,000
6	Definite	45.0	18.8	35,000,000

[a] The concentration of the product was held constant at 20° Brix during processing by the addition of pasteurized deionized water. A pan temperature of 79 to 80° F. (26.1 to 26.7° C.) was maintained.

TABLE 2

Off-flavor development, diacetyl content, and microbiological counts in pasteurized 20° Brix orange concentrate, pH 3.9, after inoculation with spoilage organism [f] and processing in evaporator [g]

Processing time	Off-flavor development	Diacetyl content Procedure A	Direct microscopic count	Plate count
hr.		p.p.m.	Average of 25 fields	Orange serum agar, pH 5.4
Run No. 4				
Initial	None	0	0.3	192,000
5	None	0.4	0.5	.....
6	None	0.0	0.4	273,000
7	None	0.5	0.5	.....
8	None	0.7	0.7	405,000
9	None	0.7	0.6	515,000
10	None	1.0	0.5	750,000
11	None	1.0	0.8	.....
12	None	1.3	0.7	1,260,000
13	None	1.5	1.2	.....
14	None	6.0	1.8	2,400,000
15	Slight	8.0	2.3	3,420,000

[f] Isolated from spoiled commercial sample of 42° Brix orange concentrate. A gum-forming, spherical organism on orange serum agar.

[g] The concentration of the product was held constant at 20° Brix during processing by the addition of pasteurized deionized water. A pan temperature of 79 to 80° F. (26.1 to 26.7° C.) was maintained.

Add 4.0 ml. KOH solution containing creatine and 10 ml. alpha-naphthol solution; stopper the test tube, shake *vigorously* for 1 min., and then allow to stand for 10 min. Shake again *vigorously* for 1 min.

Centrifuge sample for 10 min. at approximately 2000 r.p.m., then carefully decant supernatant solution into colorimeter tube and compare the color intensity of it with that of a control sample of citrus juice that has been treated in the same manner.

Determine concentration of diacetyl in test sample by using a calibration curve obtained by applying this procedure to samples of citrus juice to which known amounts of diacetyl have been added.

Procedure B. Reconstitute sample of citrus concentrate to be tested to 12° Brix ± 0.2°.

Distill 25 ml. from 300 ml. of reconstituted juice using an efficient condenser. Collect the distillate in a graduated cylinder in such a manner that any peel oil distilled off is not dispersed through the distillate, thereby causing cloudiness.

TABLE 3

Comparison of two procedures for diacetyl test for microbiological activity in concentrated orange juice [h]

Processing time	Procedure A	Procedure B	
	Reconstituted juice	Distillate	Reconstituted juice-calculated
hr.	Diacetyl—p.p.m.	Diacetyl—p.p.m.	
Run No. 5			
Initial	0	1.3	0.1
3	0	1.2	0.1
4	0.3	1.2	0.1
10	1.0	2.3	0.2
13	1.6	4.3	0.4
14	1.6	5.7	0.5
15	2.0	8.7	0.7
16	3.2	13.4	1.2
Run No. 6			
Initial	0.7	2.3	0.2
3	0.7	1.9	0.2
4	0.8	2.2	0.2
6	1.2	2.6	0.2
8	1.6	3.4	0.3
9	2.2	5.3	0.4
12	3.8	10.1	0.8
14	5.1	17.1	1.4
15	8.3	22.9	1.9

[h] Pasteurized 20° Brix orange concentrate was inoculated with *Lactobacillus plantarum* and processed in evaporator. The concentration of the product was held constant at 20° Brix during processing by the addition of pasteurized deionized water. A pan temperature of 79 to 80° F. (26.1 to 26.7° C.) was maintained.

Pipette 10 ml. of distillate from graduated cylinder, without disturbing any floating peel oil, into a 25 mm. x 200 mm. test tube.

Add 2.0 ml. of KOH solution containing creatine and 5 ml. of alphanaphthol solution, stopper the test tube, shake *vigorously* for 15 sec., and then allow to stand for 10 min. Shake again *vigorously* for 15 sec. In the same manner and at the same time prepare a reagent blank using distilled water in place of the distillate from the juice.

Fill colorimeter tubes with test sample and reagent blank and measure difference in color intensity.

Determine concentration of diacetyl in distillate by using a calibration curve obtained by applying this procedure to distilled water to which known amounts of diacetyl have been added.

Microbiological methods. *Direct Microscopic Count.* The slides were prepared in the following manner. The citrus concentrate was reconstituted to 12° Brix ± 0.2° and then 5 ml. transferred to a clean test tube. Five ml. of a filtered 0.075% aqueous solution of crystal violet, color index 681, were added to the juice. The tube was stoppered and shaken. A 0.01 ml. of the juice-dye mixture was transferred by means of a 0.01-ml. pipette to a thoroughly clean microscope slide. The material was distributed evenly over a one sq. cm. area on the slide by means of a straight nichrome wire. The slide was then placed under a heat lamp until dry. When the smear had dried, it was

sprayed liberally with Spraint or Krylon plastic spray and then dried. This plastic coating does not interfere with clear microscopic vision and produces a slide which is permanent and can be filed. No other means of fixing is necessary and there is no destaining or washing. This technique of using a plastic spray prevents juice or organisms therein from being lost during preparation of the slide, a difficulty previously encountered in making smears of citrus juices for microscopic examination. Using this procedure the microorganisms are stained a dark violet and are easily visible on the faint to light violet background. The number of microorganisms in each of 25 fields was counted and the average count per field calculated. The area of the field of the microscope used was 0.018 sq. mm.

Plate Count. Total plate counts were made by using orange serum agar, pH 5.4.

Organoleptic method. Off-flavor is detectable by the senses of taste and smell. However, detection by this method depends upon the ability of the individual to recognize specific types of off-flavor. Such ability varies from person to person, and in an individual from day to day. The indication of off-flavors in orange concentrates used in this investigation is based on the opinions of 2 persons, who were thoroughly familiar with the specific type of off-flavor produced in the products by the bacteria used. A *definite* off-flavor indicates that the concentrate was spoiled and would be recognized as such by most individuals. A *slight* off-flavor would not be detectable by most individuals, and in such cases it was believed that the product would not have to be discarded.

Pilot plant operations. The orange concentrates used in this investigation were processed in either a single-stage (1) or a 2-stage (2) high-vacuum, low-temperature pilot plant evaporator. In most of the experimental runs 20° Brix orange concentrate was pasteurized, inoculated with the spoilage bacteria, and then processed in the evaporator for a period of time sufficient to allow growth of the microorganisms. During this processing period the concentration of the product was held constant at 20° Brix by the addition of pasteurized deionized water, and a pan temperature of 79 to 80° F. (26.1 to 26.7° C.) was maintained. During each run, a sample of concentrate was withdrawn from the evaporator at hourly intervals for examination for indication of spoilage by the chemical, microbiological, and organoleptic methods.

EXPERIMENTAL RESULTS AND DISCUSSION

Data reported in Tables 1 and 2 show the off-flavor development, diacetyl content, and microbiological growth that occurred in several pasteurized 20° Brix orange concentrates during processing after they were inoculated with bacteria capable of producing spoilage.

Considering the 3 rapid methods used for detection of spoilage in the orange concentrate during processing, direct microscopic count gave the first indication of bacterial growth. Growth of the spoilage organisms was detectable by both the chemical or diacetyl test (Procedure A) and direct microscopic count prior to definite off-flavor development. However, slight off-flavor was noticeable in some instances at the same time that growth was detectable by the diacetyl test. From a practical standpoint and considering the many factors involved, there was actually no great difference between the efficiency of these three methods for detecting bacterial growth. As pointed out previously, the direct microscopic count method requires an experienced technician, and the organoleptic method involves the many differences in an individual's ability to detect in citrus juice off-flavors of a specific type.

Recently a modification of the diacetyl test (Procedure B) resulted in increased sensitivity, as is shown by data presented in Table 3. In this procedure the diacetyl is removed from the reconstituted juice by distillation, and is then more easily detectable in the clear distillate because of its greater concentration and the absence of colored substances that are in orange juice. With this increased sensitivity, it is believed that the diacetyl test (Procedure B) may be used as a rapid and reliable plant control method for the detection of spoilage of citrus concentrates by bacteria capable of producing either acetylmethylcarbinol or diacetyl.

When the diacetyl content in the reconstituted juice was calculated (Table 3) from that found in the distillate (Procedure B), it was less than that found in the juice using the original method (Procedure A). This perhaps was caused by the presence of more acetylmethylcarbinol in the reconstituted juice than would be found in the distillate, since only a portion of this compound would be removed from the juice by distillation, whereas almost all of the more volatile diacetyl would be recovered in the distillate.

Application of diacetyl test during commercial operations. The diacetyl test (Procedure B) is recommended as a rapid and reliable method for detection of growth of bacteria that cause spoilage of citrus concentrate during the first stages of concentration. Samples of concentrate should be taken from the evaporator at periodic intervals during processing and checked for diacetyl. When several consecutive tests indicate a significant increase in diacetyl, processing should be discontinued and the evaporator and other equipment cleaned, since continued operation will only result in continued spoilage. Detection of spoilage in samples from the evaporator may eliminate the packing of product of low quality, whereas if tests are only made on the finished 42° Brix concentrate, after blending of concentrate from evaporator with cutback juice, then considerable quantities of product of low quality may be frozen in cans before the trouble has been detected. Periodic checks on the finished 42° Brix concentrate should also be made as an additional control measure.

It should be pointed out that freshly extracted citrus juices may contain minute amounts of acetylmethylcarbinol, diacetyl, or similar compounds. Haagen-Smit, Hirosawa, and Wang *(3)* reported the presence of acetylmethylcarbinol in grapes. When 15 samples of fresh orange juice were distilled and the distillate checked for diacetyl (Procedure B), a range from less than 0.1 p.p.m. to 0.4 p.p.m. of diacetyl was found in the fresh juices, with an average of 0.2 p.p.m. This would correspond to a range, in the distillate, from less than 1.2 p.p.m. to 4.8 p.p.m. of diacetyl, with an average of 2.4 p.p.m. Because of these results 18 samples of frozen orange, 2 samples of tangerine, and 1 sample of grapefruit concentrate were checked for diacetyl content, and the range found in the distillate was from none to 1.2 p.p.m. This indicated a range of diacetyl from none to 0.1 p.p.m. in the reconstituted juice, which was less than that found in the fresh juices, and therefore signified some loss during evaporation.

The loss during evaporation of substances initially in fresh orange juice, which are detectable by the diacetyl test, was also confirmed by the following results. The distillate from a fresh orange juice contained 2.0 p.p.m. of diacetyl, whereas the distillate from 20° Brix concentrate made from this juice had a diacetyl content of 1.9 p.p.m.; and after this 20° Brix concentrate was processed in the evaporator for 2 hours, maintaining the ° Brix constant at 20°, the diacetyl content in the distillate was reduced to only 0.5 p.p.m.

Other applications of diacetyl test. Loss of diacetyl from spoiled 20° Brix orange concentrate during further concentration would be expected, since it is a very volatile compound. Using the diacetyl test, data were obtained that indicated an 87% loss of diacetyl from a spoiled 20° Brix orange concentrate initially having a diacetyl content of 161 p.p.m., when this product was concentrated to 70° Brix. This 70° Brix concentrate contained 21 p.p.m. of diacetyl and still had a definite off-flavor. The loss of diacetyl after concentrating the product from 20 to 60° Brix was 70%.

When off-flavors are evident in commercial citrus concentrates, the diacetyl test may be used to determine if diacetyl or acetylmethylcarbinol is present and thereby help to ascertain the cause of the off-flavor.

To see what would happen during storage at —8° F. (—22.2° C.) to the off-flavor in orange concentrate caused by the presence of diacetyl, two packs of 42° Brix orange concentrate containing 25 p.p.m. of diacetyl were prepared from juice spoiled by bacterial growth. In one pack the spoiled 55° Brix concentrate was cut back to 42° Brix with water. In the other pack the spoiled 55° Brix concentrate was cut back with freshly extracted juice. Both of the 42° Brix concentrates made from the spoiled juice had a definite off-flavor and could be easily distinguished from a control pack of 42° Brix concentrate made

from good juice. All of the packs were stored at —8° F. (—22.2° C.). Samples of these concentrates were tested periodically for 1 year using both the chemical or diacetyl and organoleptic methods. After storage for 1 year there was no significant change in the diacetyl content of either pack and each continued to have a definite off-flavor.

SUMMARY

A chemical method is described for the detection of the growth of microorganisms that cause "buttermilk" spoilage of citrus concentrate during processing. The colorimetric detection of acetylmethylcarbinol or diacetyl, which are products of bacterial metabolism, is the basis for this rapid and reliable method. Comparisons of this chemical method with other microbiological and organoleptic methods for detection of spoilage are presented.

The diacetyl content of spoiled 20° Brix orange concentrate decreased on further evaporation to 60° Brix, but a definite off-flavor was still evident in this product.

The off-flavor and diacetyl content in spoiled 42° Brix orange concentrate did not decrease significantly during storage for 1 year at —8° F. (—22.2° C.).

A technique is presented that prevents the loss of juice or microorganisms during the preparation of slides for microscopic examination.

The presence of acetylmethylcarbinol, diacetyl, or similar compounds in freshly extracted citrus juice is indicated.

Acknowledgments

The authors wish to indicate that Mr. Ellis M. Byer, Florence Foods, Inc., Florence Villa, Florida, initially suggested and used the distillation modification described in Procedure B for the diacetyl test.

The helpful assistance of Miss Betty Jo Andrews in performing some of the tests during this investigation is also acknowledged.

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Quantitative Measurements of the Organic Acids of Sorghum Sirup[a]

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(Manuscript received July 27, 1953)

A method is described for measuring quantitatively by partition chromatography the organic acids of sirup made from the common varieties of sorghum. The procedure gives complete separation of the acids. Large varietal differences in the quantities of acids present were found.

Since previous investigations of the organic acids of sorghum sirup have been qualitative, this investigation was undertaken to quantitatively determine the organic acids of sirups made from some of the common varieties of sorghum, with emphasis on varietal differences.[c]

Parsons *(6)* reported that the calcium salt of aconitic acid was deposited on the heating surface of evaporating pans used to concentrate sorghum juices which had previously been limed to neutrality. Wiley and Maxwell *(10)* working with sorghum juice considered aconitic acid to be the acid present in largest amount of those determined. They found citric, malic, acetic, tartaric, oxalic and formic acids in sorghum juice. Willaman, West and Spriestersbach *(11)* reported that oxalic, malic, citric, and tartaric acids were invariably present in the sorghum plant. Ventre, Ambler, Henry, Byall, and Paine *(7)* reported aconitic acid occurring in sorghum juice both free and combined as calcium and magnesium salts. They presented a commercially feasible method for the extraction of aconitic acid from sorghum sirup.

Organic acids can be quantitatively determined by the use of partition chromatography. Donaldson, Tulane, and Marshall *(1)* have described a chromatographic system for separation of certain organic acids using progressively changing solvents. Their system did not include a means of applying pressure which is desirable with the longer columns required to separate citric and tartaric acids. A satisfactory method of adding the required volume of sorghum sirup with sufficient acid to liberate the acids from their naturally occurring salts has not been previously reported. The procedure de-

[a] Approved for publication by the Director, Oklahoma Agricultural Experiment Station.

[b] Research assistant and professor, respectively.

[c] Except for Collier X-8-2, a promising new strain developed recently at the Oklahoma Experiment Station, the varieties used have been recommended for growing in Oklahoma.

veloped gives complete separation of the acids of sorghum sirup and quantitative results.

EXPERIMENTAL PROCEDURES

Apparatus. The chromatographic solvent system used is shown in Figure 1. The long mixing chamber (B) and the tube with a small orifice connecting it to the upper chamber (A) were used for progressive changing of the solvent. The solvents

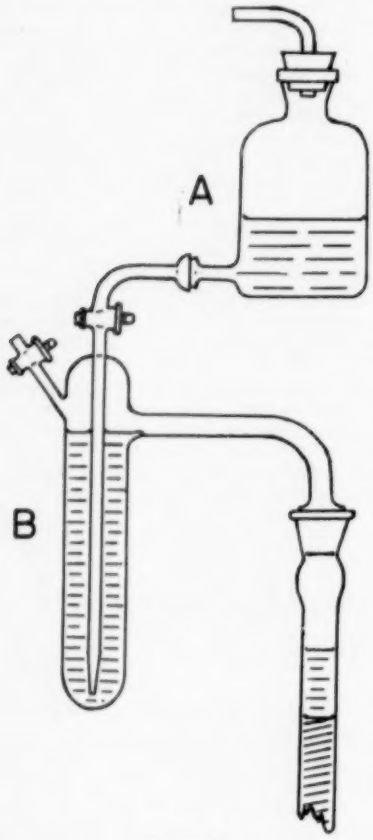

Figure 1

used were c.p. chloroform washed with water and redistilled, and c.p. 1-butanol redistilled over potassium carbonate. The mixing chamber was filled with chloroform, connected by means of the side arm to a pressure unit, brought to the desired pressure, and sealed by means of the stopcock on the side arm. The upper chamber was filled with 60% by volume of 1-butanol in chloroform. This chamber was then connected to the constant-pressure device[4] of Mader and Mader *(3)* at the same pressure already present in the mixing chamber. The connecting stopcock was then opened slightly and mixing was automatic thereafter. The collection mechanism used was that of Mader and Mader *(4)*. Fractionation of the eluent was accomplished by the use of the conductance siphon pipet of Mader and Mader *(5)*.

Preparation of samples. The sorghum cane used was grown on creek bottom land classified as Yahola sandy loam. Rows were spaced 42 inches apart, with the stalks averaging 6 inches apart. Samples were secured from 100 feet of each row, each sample representing 1/125 of an acre. The canes were stripped and headed in the field and immediately brought to a commercial mill for pressing. From this juice the sorghum sirup was prepared under laboratory conditions *(8)* using the clay clarification method described by Webster, Davies, and Sieglinger *(9)*. The samples were taken during the period when the maximum yield of sugar could be obtained for the variety. Thus, conditions under which samples were obtained closely simulated those required for commercial use.

Preparation for analysis. The column was prepared with silicic acid, Mallinckrodt's, for chromatographic analysis. Ten g. of silicic acid were mixed with 7.0 ml. of 0.5 N. sulfuric acid, slurried with chloroform, and compressed into a solid column. Two g. of pure silicic acid were then slurried, placed on top of this column, and compressed into a solid layer. Sufficient sulfuric acid was added to 25 ml. of the sorghum sirup so that when diluted to 50 ml. it was 0.5 N. A 2-ml. aliquot of this solution was forced into the upper layer of the column. After placing a few ml. of chloroform on top of this prepared column it was ready for development.

The collected 5-ml. fractions were titrated by means of a micro burette with 0.01 N. sodium hydroxide to the phenol red end point.

RESULTS

The acids were identified by their peak volume-concentrations as described by Mader *(2)*. The order of elution was acetic, fumaric, aconitic, oxalic, malic, citric, and tartaric. Table 1 shows the quantities of acids in the 5 varieties tested.

SUMMARY

The quantities of organic acids of 5 common varieties of sorghum sirup were quantitatively determined by partition chromatography. The presence of aconitic, oxalic, malic, citric, acetic, and tartaric acids was confirmed. Fumaric acid was also present. Aconitic acid was not always the predominant acid, as evidenced by Sugar Drip on an acidity equivalent basis and by Sugar Drip and Colman Y on a weight basis. In these two cases tartaric acid was predominant. Large varietal differences in the quantities of the acids present were found, but no general order of predominating acidity existed among the varieties studied.

[4] A solenoid value controlled by a manometer-switch.

TABLE 1

Organic acids of sorghum sirup (milliequivalents and mg. per ml. of sirup)

Variety	g. Juice per g. sirup	Units	Aconitic	Oxalic	Malic	Citric	Tartaric	Fumaric	Acetic	Total
Leoti	6.50	milleq./ml.	.310	.026	.070	.063	.180	.009	.004	.662
		mg./ml.	17.99	1.17	4.69	4.03	13.51	.52	.24	
Collier X 8-2	5.38	milleq./ml.	.290	.021	.036	.042	.089	.004	.002	.484
		mg./ml.	16.83	.94	2.41	2.69	6.78	.23	.12	
Colman Y	5.48	milleq./ml.	.172	.014	.036	.051	.139	.006	.004	.422
		mg./ml.	9.98	.63	2.41	3.27	10.43	.35	.24	
Sugar Drip	5.75	milleq./ml.	.084	.012	.022	.107	.174	.006	.005	.410
		mg./ml.	4.87	.54	1.48	6.85	13.06	.35	.30	
Crystal Drip	6.75	milleq./ml.	.281	.014	.059	.046	.162	.012	.008	.582
		mg./ml.	16.31	.63	3.96	2.95	12.14	.70	.48	

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Visual Detection of Either Diacetyl or Acetyl-Methyl-Carbinol in Frozen Concentrated Orange Juice

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(Manuscript received June 30, 1953)

A method is described for determining very low concentrations of either diacetyl or acetyl-methyl-carbinol in frozen orange concentrate, thereby making it possible to use this test as a control for the buttermilk odor and flavor as well as an indication of microbial growth. Methods of application to orange concentration are described.

Recently a paper was presented at the 13th Annual Meeting of the IFT by E. C. Hill, F. W. Wenzel, and A. Barreto *(3)* on the detection of acetyl-methyl-carbinol and diacetyl in orange concentrate as an indication of the growth of certain bacteria which produce off-flavors. The basis for this chemical test entails the formation of a red compound with the presence of diacetyl, creatine and alpha-naphthol. A potassium hydroxide solution is added to oxidize acetyl-methyl-carbinol to diacetyl. The intensity of this color was measured in a colorimeter to determine the total diacetyl and acetyl-methyl-carbinol concentration. In carrying out this test on orange juice it was noticed that "slight" off-flavor was noticeable before the first point of detection of these two compounds, thereby bringing about the desirability of a more sensitive test.

Since the boiling points of diacetyl and acetyl-methyl-carbinol are 88° C. and 144° C. respectively, a rapid distillation was carried out to determine the concentrations of these two compounds in the distillate and thereby eliminate three major problems. First, the problem of dealing with a highly colored juice containing large amounts of pulp and suspended matter which make visual or colorimetric detection of small color differences very difficult. Second, the problem of detecting a very small concentration of diacetyl and acetyl-methyl-carbinol thereby giving a positive test much before the point of off-flavor spoilage. Third, the problem of differentiating the individual concentrations of these two compounds as there is a direct correlation between diacetyl concentration and off-flavor. This is not true of acetyl-methyl-carbinol, unless there is a future oxidation to diacetyl. This distillation not only gives a colorless solution but also brings about a concentration of both diacetyl and acetyl-methyl-carbinol, making it possible to detect them both qualitatively and quantitatively before this point of off-flavor spoilage.

THE DIACETYL AND ACETYL-METHYL-CARBINOL TEST

Reagents

(a) *Potassium hydroxide solution containing creatine.* Dissolve 40 grams of potassium hydroxide pellets in a sufficient quantity of distilled water to make 100 mls. of solution and then add 2.0 grams of creatine (C.P.). Stir mixture until dissolved. Store at 0° F.

(b) *Alpha-naphthol-reagent.* Dissolve 5 grams of alpha-naphthol (C.P.) in 100 mls. of 99% isopropyl alcohol. Store at 0° F.

Procedure

(a) Add 300.0 mls. of reconstituted juice (12° Brix) to a large boiling flask connected to a water condenser to allow for a rapid distillation.

(b) Distill off 25.0 mls. of distillate and filter this aliquot to eliminate oil or cloud interference from high oil juice samples. Pour in a 25 mm. x 200 mm. test tube.

(c) Add 10.0 mls. of alpha-naphthol solution.

(d) Add 4.0 mls. of potassium hydroxide solution containing creatine and stopper the tube. Addition of the alpha-naphthol and potassium hydroxide in the reverse order will cause erroneous results.

(e) Shake vigorously for 30 seconds and immediately transfer a portion to a colorimeter tube and record reading 60 seconds after the reagents have been added.

(f) Wait 30 minutes and record second reading. A blank containing distilled water and the two reagents should be held for the same period at each colorimetric reading.

(g) Prepare a calibration curve by applying this method to samples of water to which known amounts of diacetyl have been added. A 550 mμ filter is used.

DISCUSSION

The diacetyl color reaction is almost complete after 60 seconds, whereas the acetyl-methyl-carbinol color reaction has just begun if the latter compound is not present in concentrations over 6-8 p.p.m. in the distillate sample. This can be regulated by diluting the juice with distilled water and the final results converted back to the desired concentration. This increase in color over a 30-minute period is illustrated in Figure 1. Therefore, the one minute reading represents the diacetyl content of the distillate and the difference between the one minute and 30 minute readings represents the acetyl-methyl-carbinol content.

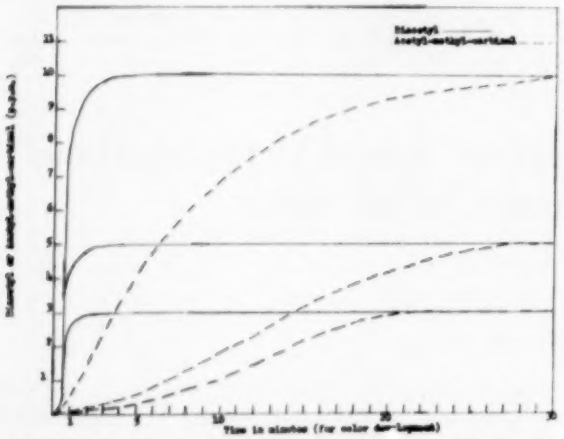

Figure 1. Color development of diacetyl and acetyl-methyl-carbinol in various concentrations.

If a qualitative test is desired a visual observation of the red color after one minute is a positive test for diacetyl, and the increased color formed afterwards is an indication of the presence of acetyl-methyl-carbinol. A positive test does not necessarily denote off-flavor but merely a condition which may lead to off-flavors, depending on the individual concentrations of these two compounds, microbial growth, and other conditions present at that time.

During the distillation of juice the diacetyl and acetyl-methyl-carbinol are concentrated approximately eight-fold and two-fold respectively. If the distillate should give a positive test of 3.0 p.p.m. of diacetyl after 60 seconds and 5.0 p.p.m. of diacetyl and acetyl-methyl-carbinol after standing 30 minutes, there is 3 p.p.m. of diacetyl and 2.0 p.p.m. of acetyl-methyl-carbinol in the distillate. This would indicate approximately 0.4 p.p.m. (3.0 p.p.m. divided by 8 because of concentration) of diacetyl and 1.0 p.p.m. (2.0 p.p.m. divided by 2 because of concentration) of acetyl-methyl-carbinol in the orange juice sample.

In juices with very high concentrations of acetyl-methyl-carbinol a comparison of the first and third 25-ml. aliquot of distillate after the 30-minute period will give an indication of the concentrations of these two compounds. Since there will be no diacetyl in the third aliquot and the acetyl-methyl-carbinol decreases only slightly with each successive portion, the third aliquot approximates the acetyl-methyl-carbinol, and the difference between the two approximates the diacetyl concentration.

For a general control measure the relative change of concentration of these 2 compounds in juice at various processing points is a good indication of plant sanitation. The points usually selected are the cut-back juice, evaporator feed, 20 Degree Brix stage, and the final product. As the juice passes through the evaporation system to a higher concentration, the diacetyl and acetyl-methyl-carbinol concentrations decrease rapidly because of the partial evaporation of these compounds. The 20° Brix stage of the evaporation system is known to have the greatest growth of gum former organisms which in turn may produce diacetyl. Some of the investigators responsible for this work on 20° Brix juice are Hays and Riester *(2)*, M. J. Byer *(1)* and Murdock, Troy and Folinazzo *(4)*. The acetyl-methyl-carbinol and diacetyl level should be determined on these selected points in the system and checks made continuously to detect any changes that may take place. A comparison of colorimetric readings at various points in the process is all that is necessary for this control measure rather than a conversion to actual p.p.m.

The individual concentrations of these two compounds are very useful in determining not only the present degree of buttermilk off-flavor spoilage due to diacetyl but also the potential off-flavor spoilage due to acetyl-methyl-carbinol, since the latter compound can be oxidized to diacetyl or reduced to 2, 3-butylene glycol depending upon the conditions present. There seems to be no correlation between the individual concentrations of diacetyl and acetyl-methyl-carbinol in off-flavored juices. Various amounts of acetyl-methyl-carbinol can generally be found in normal orange juice samples but only the slightest traces of diacetyl have been detected. This test is intended to be a quality control measure and not a strict quantitative analysis.

SUMMARY

A method has been described for determining very low concentrations of either diacetyl or acetyl-methyl-carbinol which are compounds that may lead to the buttermilk odor and flavor in orange juice. Thus it is possible to measure the quantities of either of these two compounds in such minute amounts so as to allow ample time to prevent their buildup and subsequent off-flavor spoilage.

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Sugar Hydrate Formations in Frozen Citrus Concentrates[a, b]

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(Manuscript received July 25, 1953)

The undesirable change in appearance and texture caused by the formation of sugar hydrates in frozen grapefruit concentrates and limeades has presented a major problem to the citrus industry. To obtain much-needed background information on this new problem, 46 exploratory packs of these products were prepared and stored during the 1951-52 season to determine the effect of different sugars, heat treatment, and packaging on these formations. Results are discussed in the present paper.

The Florida Citrus Code of 1949 *(4)* prohibits the addition of sugar to Florida frozen concentrated orange juice. However, frozen grapefruit concentrate may be produced either with or without added sweetening ingredient. Furthermore, there is the likelihood of increased production of frozen limeade which has a higher sugar content. In the past, the industry has encountered some difficulty with sugar-added frozen grapefruit concentrate due to the formation of sugar hydrates, which are likely to appear under certain conditions. These sugar hydrates first appear on the surface of the concentrate (Figure 1) in the form of small white

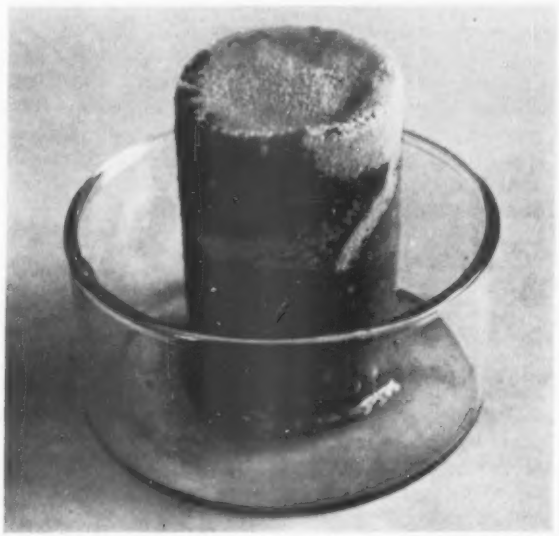

Figure 1. Sugar hydrate formation on frozen grapefruit concentrate.

particles which, in the case of dextrose, are waxy in texture and have low solubility. This is not a new problem to the food industry, but Young, Jones, and Lewis *(6)*, who have done much of the fundamental work with sugar hydrates, point out that investigations must be carried out on each specific product before application to commercial operations. Since little work has been done concerning sugar hydrates in these citrus products, it was deemed advisable to present this discussion of the possible factors causing their formation and to report the results obtained from examination of some exploratory packs.

This investigation was concerned only with possible factors causing sugar hydrate formation that can be controlled by the manufacturer. The major controllable causes for this formation are the type and quantity of added sugar. Brekke and Talburt *(2)* encountered greater hydrate formation with dextrose than sucrose. The general consensus of manufacturers of frozen fruits *(3)* was that dextrose in excess of 25 to 33% of total sweetener agent caused crystallization (presumably, the hydrate). Dextrose appears to be a major factor in hydrate formation and, because of its higher melting point and lower solubility *(2)*, could be more of a problem than sucrose. The majority of the exploratory packs were put up with this in mind. Naturally, the period and temperature of storage *(2)* of these products play an important role in this formation. The possible effect of heat treatment in the manufacture of frozen grapefruit concentrate was also considered. The possibility of undissolved sugars (seeding) in these products, particularly limeade with its higher sugar concentration, was studied. The final factor investigated was the effect of using non-hermetically sealed containers.

EXPERIMENTAL PROCEDURE AND RESULTS

Grove-run Duncan grapefruit and Persian limes were the only citrus fruits used. Most products were prepared in the same manner as that done commercially, using the pilot plant at the Citrus Experiment Station. Since this was exploratory work with numerous variables, it was impossible to follow in all cases exact commercial practices. The grapefruit was given a normal washing, then extracted with a Skinner rotary press, and the juice passed through a Food Machinery finisher (0.030-inch screen). A Citro-Mat extractor was used for the limes. The grapefruit concentrate was prepared in the Atkins evaporator *(1)*. For most packs, the juice was concentrated to about 55° Brix and then cut back with freshly extracted grapefruit juice to 36° Brix and sugar added. U. S. D. A. Standards *(5)* require that if this product is to be sugar-added, it must contain at least 36° Brix natural juice solids (38° to 42° Brix for unsweetened), and sugar added to finish out at between 38° and 48° Brix depending on its Brix/acid ratio. The final products did not necessarily meet these standards.

Effect of added sugar composition. The sugars added consisted of sucrose (commercial cane sugar) and dextrose (Cere-

TABLE 1

Effect of added sugar combinations on formation of sugar hydrates in frozen grapefruit concentrates

Sample	Sugar added to raise Brix from 36° to 48°		Rate of hydrate formation at −8° F. (−22.2° C.)			
	Sucrose	Dextrose	Initial	6 mo.	9 mo.	12 mo.
	%	%				
1	100	0	None	None	None	None
2	90	10	None	None	None	None
3	80	20	None	None	None	None
4	70	30	None	None	None	None
5	60	40	None	None	Slight	Slight
6	50	50	None	Medium	Medium	Extreme

[a] Presented at the Thirteenth Annual Meeting of the IFT, Boston, Massachusetts, June 23, 1953.

[b] Cooperative research project of Florida Citrus Experiment Station and Florida Citrus Commission. Florida Agricultural Experiment Station Journal Series, No. 175.

[c] Florida Citrus Experiment Station.

[d] Florida Citrus Commission.

TABLE 2
Effect of heat treatment on formation of sugar hydrates in frozen grapefruit concentrates[a]

Sample	Treatment of juice	Rate of hydrate formation at —8° F. (—22.2° C.)				
		Initial	3 mo.	6 mo.	9 mo.	12 mo.
8	Sugar added, heat treated, concentrated, and unheated sweetened cut-back juice added.	None	None	None	None	None
9	Heat treated, concentrated, and sugar added in unheated cut-back juice.	None	None	Slight	Medium	Extreme
6	Concentrated and sugar added in unheated cut-back juice.	None	None	Medium	Medium	Extreme
7	Sample 6, seeded	None	Medium	Medium	.....	.....

[a] Sugar used in all cases was a mixture of 50% sucrose and 50% dextrose.

lose assumed to contain 9% water of crystallization) which were dissolved in the cut-back juice. A total of 22 packs was prepared, and a typical series of 6 is shown in Table 1. For brevity, data are presented only for this series, as the data from the other packs merely confirmed these results. Although all packs were examined monthly, only the results after storage for 6, 9, and 12 months are shown and discussed.

When the percentage of dextrose in the added sugar mixture was increased beyond 30%, the hydrate formation appeared and similarly increased. However, it might be mentioned that the percentage or total amount of dextrose does not appear to be the sole factor in this formation. In a limeade pack containing added dextrose corresponding to 20% of total solids, no hydrate formed after 18 months' storage at —8° F. (—22.2° C.); whereas, in a grapefruit concentrate (sample 6) containing added dextrose corresponding to 12½% of the total solids, hydrate formation readily appeared after 6 months.

Effect of heat treatment. The effect of various temperatures of heat treatment was not studied, but a relatively high temperature for frozen concentrate was used. The single-strength juice was passed through a tubular heater and heated to 195° F. (90.6° C.) in about 13 sec. and then cooled in about 9 sec. to 80° F. (26.7° C.). The cut-back juices were not heated. The effect on hydrate formation when sugar is added to the product during processing is shown in Table 2.

Only one series of packs was prepared to determine whether heat treatment showed any marked effect on hydrate formation. Data in Table 2 show that when sugar was added before heat treatment no hydrate appeared during a 12-month storage period, whereas, when the sugar was added after heat treatment, the hydrate readily appeared. It is doubtful if this can be attributed to undissolved sugars since seeded samples readily showed hydrate formation as in the case of sample 7. Further work must be carried out to determine the factors causing this retarding of hydrate formations.

Effect of seeding. Sugar crystals (50% dextrose and 50% sucrose) were added to 17 packs to have controls for accidental seeding or undissolved sugars in the final product. The seeded samples in some instances formed the hydrate, whereas the unseeded did not; in some samples seeding hastened the formation by from 3 to 6 months. There is little point in going into further detail concerning the results obtained as the hastening action of seeding on crystallization is well known.

Effect of packaging. The majority of the packs were vacuum packed in tin cans. Hydrate formation in one set of grapefruit concentrate that was packed in waxed paper containers (non-hermetically) was hastened over that of the corresponding hermetically sealed cans by from 3 to 6 months.

DISCUSSION

Since this work was of a practical nature, the grading of hydrate formation was also done with this in mind; an "extreme" grade was considered as being objectionable to a consumer, a "medium" grade as being definitely noticeable, and grades of "slight" and "questionable" as not being of consumer significance. The hydrate formation usually starts at the bottom of the can and gradually works up the sides and towards the center. Eventually, in extreme cases, the product has either a thick crust over the entire surface or sometimes the whole product is a solid mass of crystals.

Because seeding has been shown to play such a major role in hydrate formation, it might be well to caution manufacturers to be extremely careful not to leave any undissolved sugars in their product. Results indicate that the use of hermetically sealed containers is necessary. Products must also be moved from cold storage as rapidly as possible and in chronological order since the longer the storage the more likelihood of hydrate formation.

The temperature of storage and temperature variations during storage were not studied in this investigation. Also not investigated were other uncontrollable factors from a practical viewpoint—factors such as pH, maturity, viscosity, surface effects, and the undependability of spontaneous nucleation. However, as a result of these preliminary findings, 147 packs were processed and are now in storage; they will be used to investigate some of the questions not yet answered that have arisen from this work.

SUMMARY

Exploratory packs of frozen grapefruit concentrates and limeades were prepared containing various amounts of sucrose and dextrose. These were examined monthly for sugar hydrate formations during storage at —8° F. (—22.2° C.). It was found that amount and ratio of added sugars, heat treatment, seeding, type of container, and length of storage all played an important role in sugar hydrate formation. As a result of this work, these factors and others are being further investigated.

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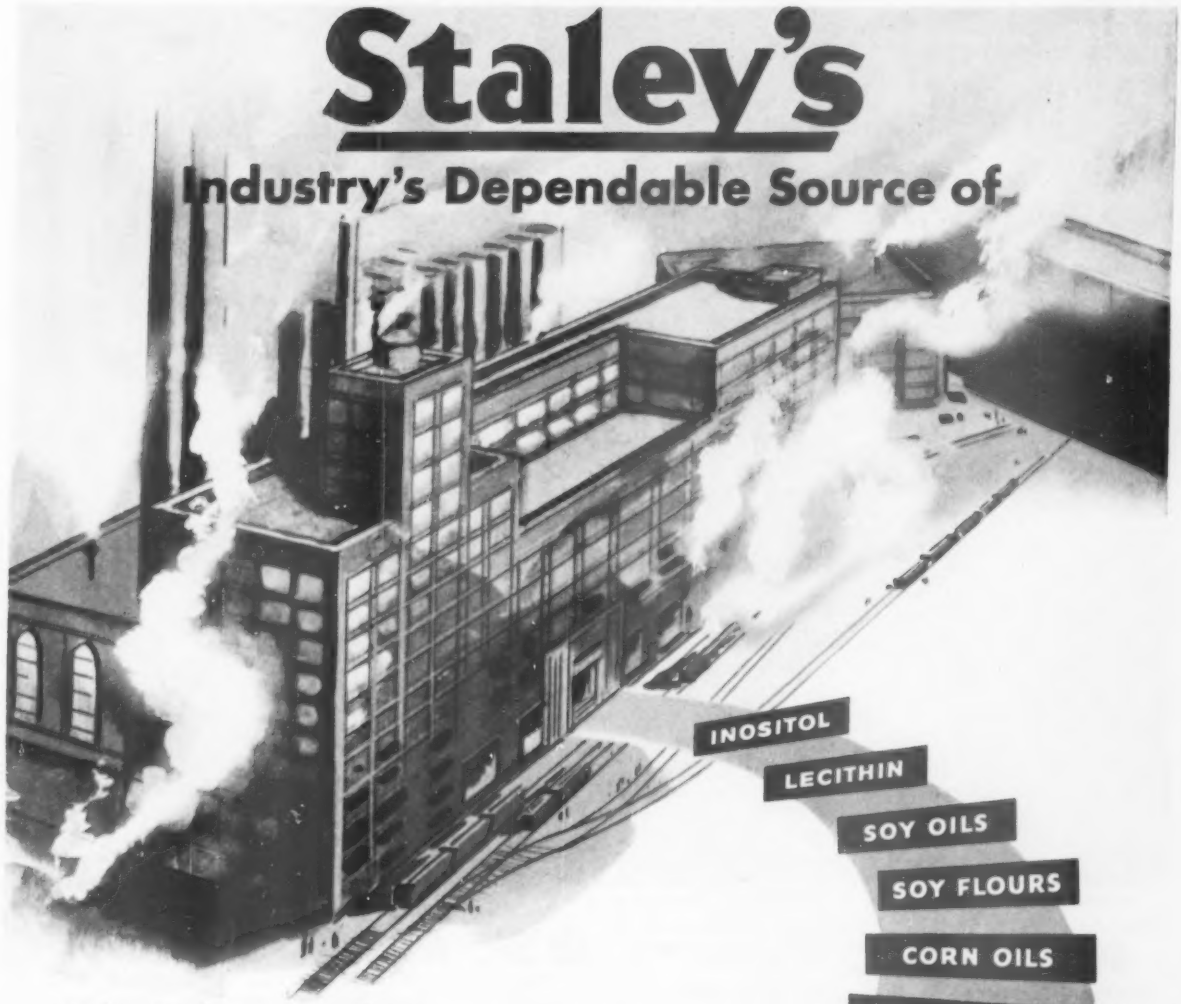

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As selling becomes more competitive...the food processor must prove to the housewife the superiority of his product.

The most important factor in building "brand preference" is better flavor. The housewife is faithful to the brand that tastes best to her and her family.

Here's how to insure a "flavor edge" over competition for your product.

NO MATTER what type of food you process, its most crucial test is at the consumer's table. Advertising and salesmanship may account for the first sale. But with many brands competing for the shopper's food dollars, only superior flavor will make her a steady customer.

No wonder, then, that so many leading food processors are *insuring* maximum flavor in their products with Ac'cent. For Ac'cent brings out and holds *all* natural flavors from processor to consumer. Ac'cent is monosodium glutamate in its purest, most convenient form. When added to foods during processing it restores nature's essential flavor factor of protein...glutamate...the protein factor that fades during shipping, storage and cooking. Ac'cent enhances the fresh, delicious flavor of foods...without adding flavor of its own.

In these days of increasingly competitive selling, make sure your brand has a "flavor edge." Ac'cent service staff will be glad to arrange a test pack so you can discover for yourself what an amazing difference Ac'cent will make in your product. And they will make suggestions on the most economical way of adding Ac'cent to your operation.

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PURE MONOSODIUM GLUTAMATE
...makes processed food taste better!

AMINO PRODUCTS Division of International Minerals & Chemical Corporation, 20 North Wacker Drive, Chicago 6, Illinois.

SELECTED ABSTRACTS

(Continued from page 18 preceding technical papers)

hyde-tanned leather powder and powd. silk, hair, and Capron (polyamide fiber). Most of the natural products lost their hydrophobic protective film during the grinding process, so that they collected in the aq. phase after grinding. Only with the polyamide fiber was this effect not observed.

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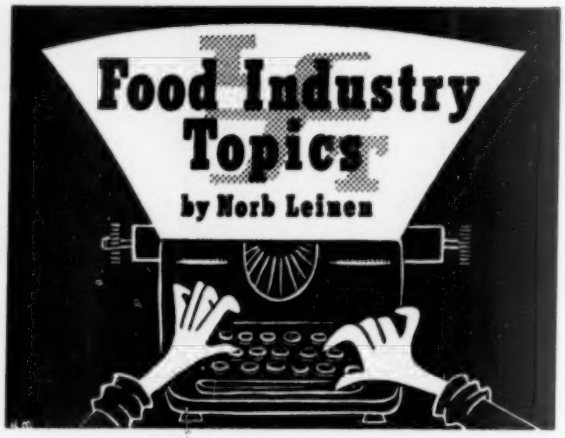


. . . *in the public interest*

The policies with regard to **addition of specific nutrients to foods,** adopted in 1941 by the Food and Nutrition Board of the National Research Council, have recently been reaffirmed in principle by the Food and Nutrition Board and the Council on Foods and Nutrition in a "joint statement of general policy in regard to the addition of specific nutrients to foods." However, use of natural foods in meeting nutritional needs as far as practicable is emphasized.

◆ ◆ ◆

A new program of **research on fatty acids,** sponsored by the Fatty Acid Division of the Association of American Soap and Glycerine Producers, Inc., New York City, was announced in January. It will be carried on in cooperation with the Utilization Research Branch, Agricultural Research Service, USDA, at the Eastern Regional Research Laboratory, Philadelphia. The initial area of study will be "A Study of Lesser Known Addition and Substitution Reactions Involving Fatty Acids (unsaturated and saturated) with Particular Reference to Compounds of Nitrogen, Sulfur, and Phosphorous."

◆ ◆ ◆

Nearing completion is the new cereal technology building of the North Dakota Agricultural College, Fargo. In addition to housing milling, baking, and macaroni processing equipment, the new structure—a veritable "research center for grains"—will include physical dough testing apparatus, facilities for evaluating the malting quality of barley varieties, and the various scientific instruments required for advanced research in the cereal field. Besides investigations directed toward the improvement of wheat and barley, the Dept. of Cereal Technology, under the direction of Dr. R. H. Harris, conducts investigations on cereal proteins, starches, enzymes, and on properties of wheat and barley that are related to baking performance.

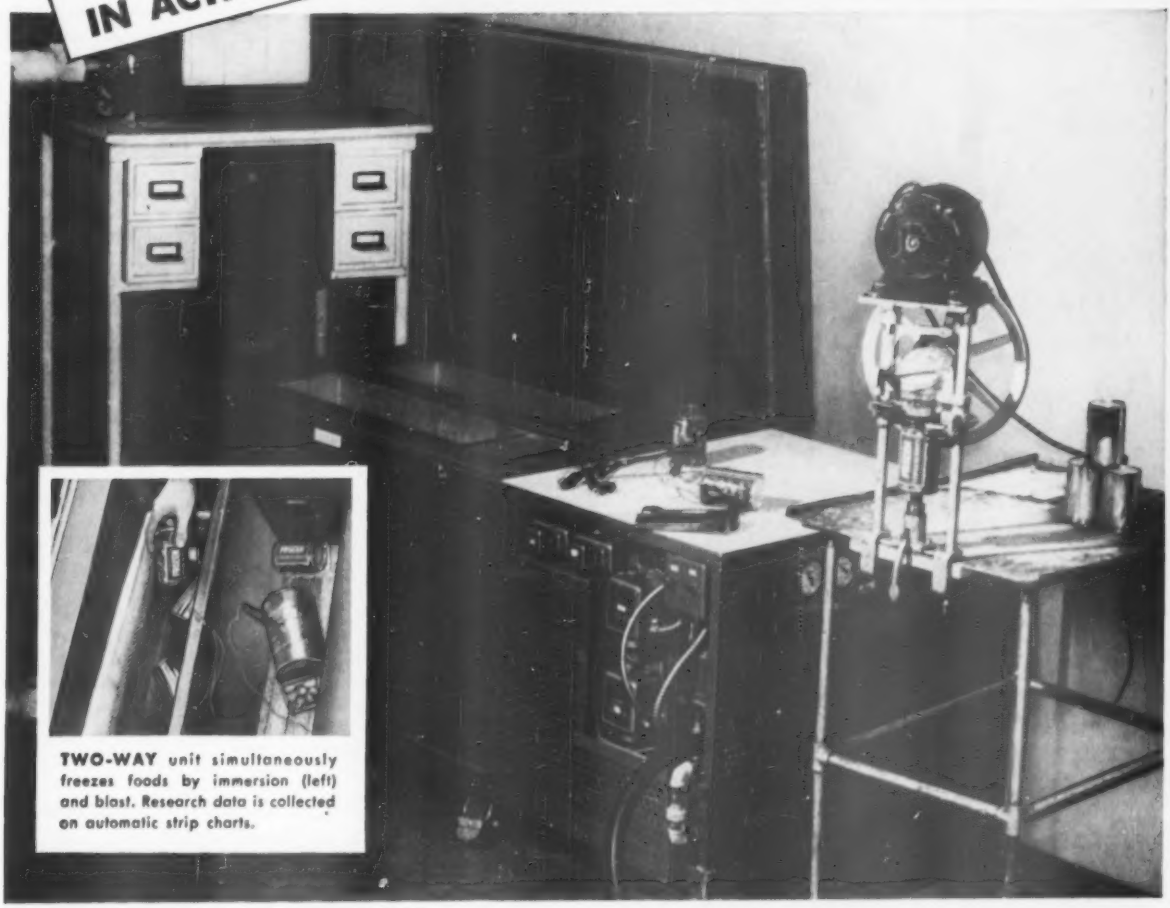
◆ ◆ ◆

The average American ate 44% better in 1953 than he did prewar, said Mr. Wm. J. Searlett, food industry manager for the Industrial Division of Minneapolis-Honeywell Regulator Co., Philadelphia, and an IFT member. He attributes the

(Continued on page 26)

Continental **RESEARCH IN ACTION**

New portable laboratory speeds answers to freezers' problems



TWO-WAY unit simultaneously freezes foods by immersion (left) and blast. Research data is collected on automatic strip charts.

LAB EQUIPMENT includes a recording potentiometer, thermocouples, 2-way freezing unit, and atmospheric and gas-vacuum closing machines.

In the interest of its can-using customers, Continental has long maintained 17 field research laboratories throughout the U. S. Now it has an "18th"—a compact, portable laboratory which travels right into the plants of frozen food packers.

Quickly set up in 48 square feet of space, it carries out in miniature the packaging and freezing operations of a full-size freezing plant. Whether the product to be packed is a fruit, a vegetable or a seafood, the portable laboratory will help pick the best freezing time, the best size and shape of can to use, and the best packing procedure — air closure, vacuum or gas pack.

Continental's portable laboratory was designed and built by its own scientists and engineers. It has already proved its worth by helping several of our customers in widely separated sections of the country to produce better products at lower cost... and in helping Continental produce tailor-made containers for a fast-growing industry.

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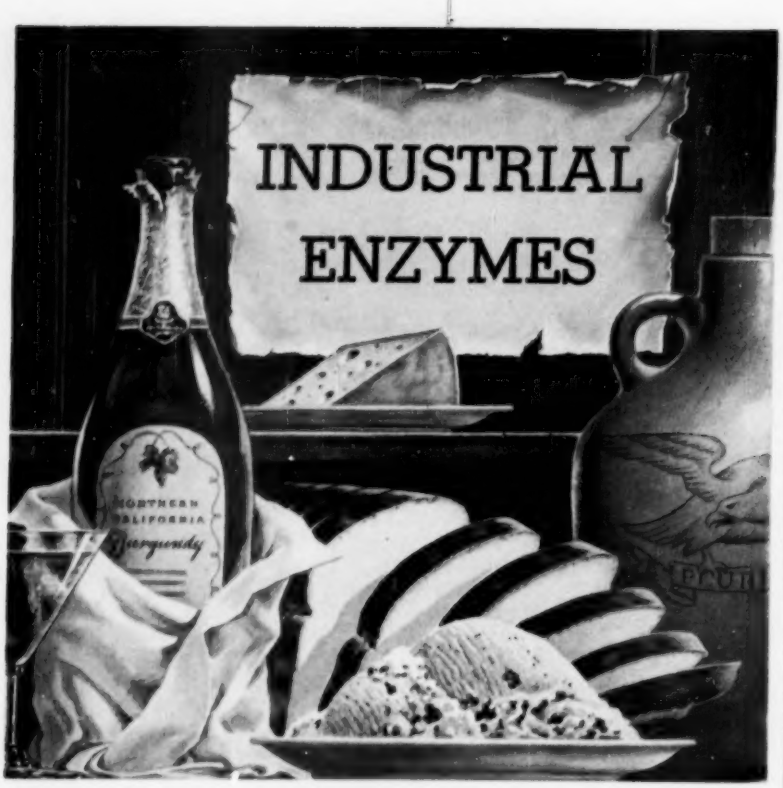

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Rohm & Haas industrial enzymes are valuable tools for the food technologist. Besides their long-established use in wine making and fruit juice clarification, they've become important in other ways: they tenderize meats, improve texture and grain of bread, produce sweet syrups from corn starch. They also permit the use of higher concentrations of skim milk in ice cream without danger of "sandiness".

In contrast to many chemicals, Rohm & Haas industrial enzymes are *natural* products, which catalyze *specific* reactions and prevent undesirable side-reactions. A little enzyme goes a long way toward increased process efficiency, toward big gains in sales appeal.

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CHEMICALS FOR AGRICULTURE

ROHM & HAAS COMPANY

WASHINGTON SQUARE, PHILADELPHIA 5, PA.

Representatives in principal foreign countries

FOOD INDUSTRY TOPICS

(Continued from page 24)

greatly improved dietary standard to superior manufacturing and processing efficiency resulting from more general use of a multiplicity of **automatic control, measuring, and recording instruments.** Listed among the processing operations which have recently benefited from application of electronic controls are: aseptic canning, butter making, bread baking, sugar refining, smokehouse operations, and processing of concentrated frozen citrus juice. As the new processing trends take hold, he predicted that the automatic control share of total plant investment will, by 1964, be 3 to 5 times the present total which has itself increased by twelvefold within the past decade. "At that time," he states, "we will be measuring and thus controlling some of the intangibles such as color, flavor, odor, consistency, and perhaps even bacterial content. Food processing trends such as these will markedly alter the shopping habits of the American housewife."

◆ ◆ ◆

Macaroni products again appear on the brink of a record year according to Mr. Robert M. Green, secretary of the National Macaroni Institute. Estimated total sales volume for the industry in 1953 is set at $243,930,000—a $15,970,000 increase over the previous year. The majority of the nation's macaroni manufacturers expect the industry to maintain or increase this sales volume in 1954, Mr. Green predicted.

◆ ◆ ◆

Another **big year for all major branches of the chemical industry** was predicted recently by Mr. Allan P. Stern, president of the Colonial Iron Works Co., 17643 St. Clair Ave., Cleveland, O. Basing his estimate on changes in the volume of activities in the organic and inorganic chemical, pharmaceutical, plastic, petrochemical, and related industries as reflected in the Colonial sales office, Mr. Stern stated: "The family of chemical industries is undergoing such rapid technological changes, and research is creating new products so rapidly, that obsolescence of process equipment is a significant factor in creating a continued demand for newly designed types of plant facilities and equipment. Such a demand should continue long after activity in other basic industries has subsided."

. . . *things new under the sun*

Drying, heating, baking, cooking, pasteurizing, and many other industrial applications can be performed with economy, speed, and efficiency using new "Infralite" Radiant Energy Heating Lamps, according to announcement from Thermex Company, Inc., 533-535 Bergen St., Harrison, N. J. The uniformly dis-

(Continued on page 28)

there's a
reason for reaching!
frozen foods
packed with

PFIZER ASCORBIC ACID or ASCORBIC-CITRIC MIXTURES

● When housewives reach for a specific brand of fruit or fish time-after-time, there must be a reason. Behind that preference is usually a better product. One way to keep them reaching for *your* brand is to use either Pfizer Ascorbic Acid or Ascorbic-Citric Mixtures to improve the taste and appearance of your packs.

In fruits... Ascorbic Acid has been successfully used for many years to retard browning and flavor loss in peaches. Numerous packers are achieving the same outstanding results in treating cherries, apricots and apples.

For fish... Ascorbic Acid prevents "rusting" of the fatty portion... preserves natural colors ...cuts atmospheric oxidation, source of rancidity and strong, unpleasant taste. In both fruits and fish, Pfizer Ascorbic-Citric Mixtures give the same effective control of oxidation *at much lower cost.* For more information about Pfizer antioxidants call or write...

CHAS. PFIZER & CO., INC.

630 Flushing Ave., Brooklyn 6, N. Y.
Branch Offices: Chicago, Ill.; San Francisco, Calif.
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Manufacturing Chemists for Over 100 Years

Pfizer

extend SHELF LIFE!

retard RANCIDITY!

...on High-Fat Low-Moisture Foods!

● As you know, potato chips, prepared mixes, nuts, baked goods, powdered soups and many other products that are high in fat and low in moisture are often spoiled by rancidity and the development of oxidative "off" flavors. And these factors reduce the shelf life and profit margin of many food products.

These problems may be avoided by the proper use of a new food grade salt with antioxidant, developed as a result of years of laboratory research—Diamond Crystal Salt with ANTIOXIDANT!

New Diamond Crystal Salt with *Antioxidant* is as simple to use as common table salt. If sodium chloride is used in your processing, this new salt will fit into your operation without extra equipment or labor.

Diamond Crystal Salt with *Antioxidant* is economical, too! The extra cost involved in salting potato chips is only about 7¢ per 100 lbs. of chips! This is a small cost to minimize returned goods.

The effectiveness, ease of use and economy ... this new salt will more than justify its test in your operation. Samples are available upon request. Send for yours today!

Write to Technical Director, Diamond Crystal Salt Co., St. Clair, Michigan.

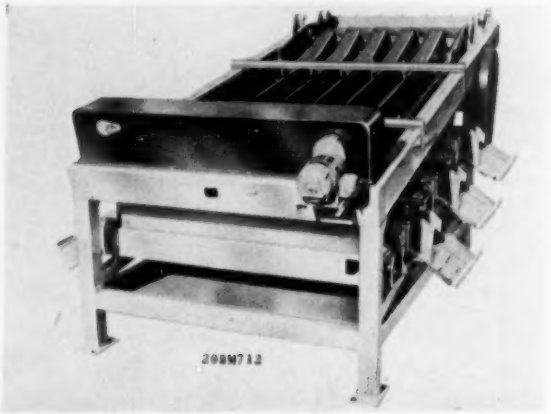
DIAMOND CRYSTAL SALT with Antioxidant

FOOD INDUSTRY TOPICS

(Continued from page 26)

tributed radiation of 10 to 200 watts per square inch of lamp surface eliminates hot spots and provides deep penetrating heat up to 2000° F. This, the manufacturer claims, is twice the possible temperature of existing metallic tubes. The life of Infralites averages about 5000 service hours. The lamps are available in a wide range of styles and sizes. Write Thermex for free descriptive literature.

◆ ◆ ◆



Molded fiberglass shaker pans, combining clean-surfaced functional beauty with stability and strength, replace the stainless steel units on these product-grading machines. The grader which permits high capacity size-grading of such diversified products as shrimp, soft figs, dill pickles, carrots, sprouts, fruits and nuts, was formerly equipped with hand fitted shaker and drain pans. Rigid testing confirmed that the porcelain-like surface of molded fiberglass does not transfer, absorb or impart undesirable effects; improves product handling while materially reducing maintenance and replacement costs to user. The size grader and molded pans are produced by the Dudley Machinery Corporation, Mountain View, California.

◆ ◆ ◆

Iron Tetrine, the iron complex of ethylenediamine tetraacetic acid, **cures and prevents iron deficiencies in agricultural crops** according to Glyco Products Co., 26 Court St., Brooklyn 1, N. Y. This iron compound has reportedly produced sensational results in curing the disease in citrus crops in the Florida acid soils. Tests and experience have shown that benefits can also be obtained in other crops and flowering shrubs.

◆ ◆ ◆

A new, **high-speed food filler** that occupies a minimum of space is now in operation at the Luzea Ravioli Co., San Francisco, for filling a variety of liquid or semi-solid Italian foods. Known as the Simplex and manufactured by F. L. Burt Co., 571 Seventh St., San Francisco 3, Calif., this semi-automatic filler fills 20 to 40 Seal-Rite cups per minute.

◆ ◆ ◆

A dramatic new rose note, of particular interest in the present market, has been sounded by Dodge & Olcott, Inc., 180 Varick St., New York City, with Rose Bulgare Synthetic. This newly created specialty, definitely rose in character, is intended as an **extender for the scarce and costly natural Rose Bulgare.** D&O suggests individual testing with trial quantities of the new product.

◆ ◆ ◆

In an effort to **decrease maintenance costs and speed the plant cleaning process,** many plant engineers have turned to the wet blasting process. This process consists of throwing water-suspended abrasives at the work by compressed air. It is particularly appropriate for cleaning problems where holes, pockets, and other recesses make it difficult to reach the contaminants and remove them. Cleaning is rapidly accomplished, and all areas on a part receive the same uniform finish. For further information on this practical process contact American Wheelabrator & Equipment Corp., Mishawaka, Ind.

(Continued on page 30)

IT'S NOT A MATTER OF

- CITRUS OILS
- TERPENELESS OILS
- AROMATICS
- ESSENTIAL OILS
- BASIC FLAVOR NOTES
- VANILLAS

The success of our products has come from laborious, meticulous laboratory development, followed by time-tested application by all types of flavor users.

Build your business with...

- The true uniformity and superb quality of Felton's essential oils, basic flavoring materials and vanillas.
- The fully characteristic notes of Felton's fine aromatics.

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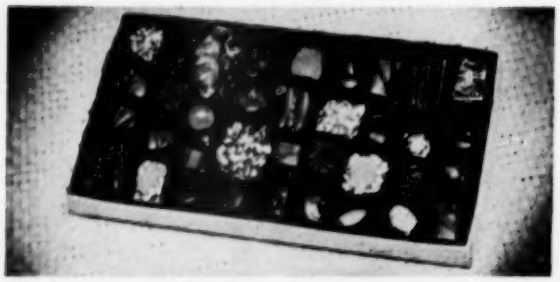
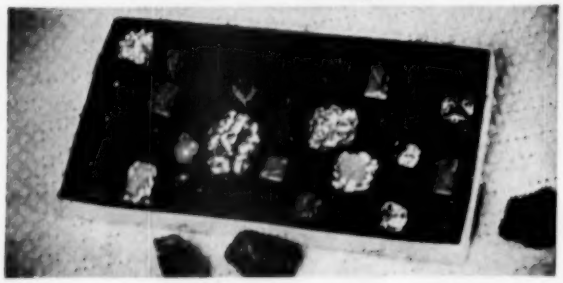
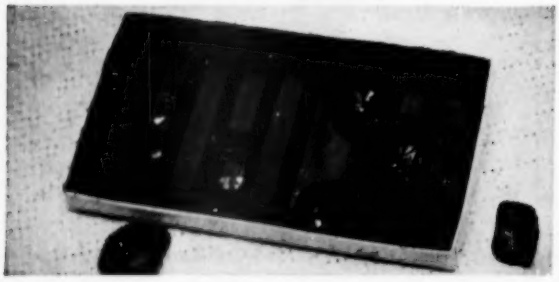
for flavors



that bring



them back



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FOOD INDUSTRY TOPICS

(Continued from page 28)

A new Feon filter fabric of polyethylene has been announced by Filtration Fabrics Division of Filtration Engineers, Inc., 155 Oraton St., Newark 4, N. J. Woven of polyethylene monofilaments, the new cloth has a relatively open mesh and is recommended principally for **free-filtering applications** such as coarse crystalline materials or fibrous pulps. The cloth is odorless, tasteless, non-toxic, and resistant to alkalis, inorganic acids, and many organic chemicals.

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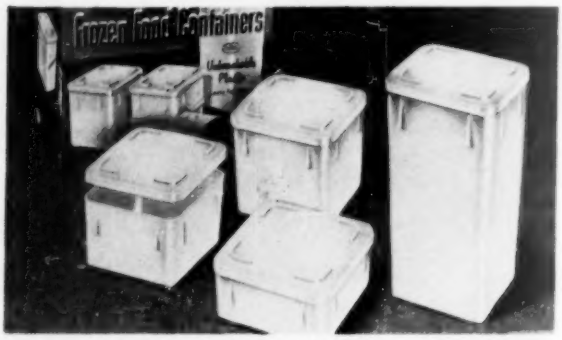


To save valuable freezer space and make home food freezing easier, Columbus Plastic Products, Inc., Columbus, O., offers a new line of uniform shaped Lustro-Ware Frozen Food containers. All 4 practical sizes from 1 pint to 2 quarts have the same square base dimension so that they stack in any combination without wasted space. Equally important is the fact that one cover fits all sizes. Molded of flexible polyethylene, these containers can be used over and over without danger of breaking.

. . . *the literary corner*

Materials accounting, continuous feeding, batch control, and continuous proportioning control are some applications of Merchen Continuous Weighing Equipment described in a new Wallace & Tiernan publication, "The Best Weigh is the Merchen Weigh." Flow diagrams, descriptions, and features of both Merchen Scale Feeders and Meters are given. Illustrations include typical installations in many different industries. Copies can be secured by writing the firm's Feeder Dept., Box 178, Newark, N. J. Ask for Publication TA-1002-M.

◆ ◆ ◆

Techniques for **converting a continuous tone image into a line drawing** by means of a new photographic technique developed by the Eastman Kodak Company are described in a new booklet, "Line Effects from Photographs by the Kodak Tone-Line Process." Free copies may be obtained by writing the Sales Service Division, Eastman Kodak Company, Rochester 4, N. Y.

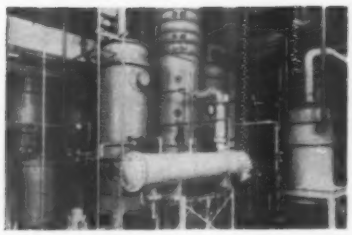
◆ ◆ ◆

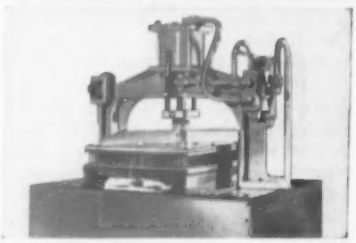
Seventeen successful new "Teflon" applications in addition to those reported in an earlier edition of March 1953 are listed in a new 12-page technical bulletin on "Teflon" tetrafluoroethylene resin finishes. Discussed are various finishing systems of "Teflon" primers and enamels over metallic and non-metallic surfaces, application methods, the fusing operation, precautions, and handling procedures. For a copy write Du Pont Finishes Division, Room D-7145, Wilmington 98, Del.

◆ ◆ ◆

A comprehensive **discussion of dispersion problems in industry and chemical processes** is found in a 40-page illustrated publication of the Kinetic Dispersion Corp., 185 Botsford Pl., Buffalo 16, N. Y. The subject is approached from both the practical and theoretical viewpoints, and in addition to studies of the behavior of materials in solid, liquid, and dispersional phases, the book contains comparison charts of results of vari-

(Continued on page 32)





Griffith Research Presents a Challenge and Big Opportunity to any Vigilant Buyer

Of special interest to you and every curing foreman, sausage maker, production superintendent, sales manager, treasurer, vice president and president.

WHATEVER his title, your purchasing agent plays a vital role in advancing the consumer popularity of your company's products. Ever searching for higher quality and better value in materials, he must be on the alert for—and inform other key men in management of—significant research developments.

The veteran buyer knows: *Rarely does research introduce an ingredient or process of revolutionary character.* His constant hope and search is for the slight improvement, another refinement, a series of steady gains. From such, your products gain an extra advantage in competitive quality or value, or both. Yet, it is the rare research development which is always a challenge and big opportunity for him.

Serving buyers in both respects, Griffith has a record unique in food processing research. Persistent, progressive, and highly diversified in character.

Griffith research is recognized for improvements in the quality and quality control of staple ingredients. For bettering food flavor. And, for reducing the time and cost of processing foods in which Griffith materials are used.

Among recent revolutionary developments are Griffith's PENETRONIC* process of "dry curing" bacon in just three days . . . Griffith's exclusive process of Spice Extraction, which controls the potency and uniformity of flavor in PEPPEROYAL** and SOLUBLIZED SEASONINGS . . . and Griffith's new Monoglyceride Antioxidants, the 100% oil-soluble stabilizers for lard and other edible fats.

These, and a score of Griffith products, will reward the vigilant buyer who looks to the food industry supplier that has the staff, experience and facilities for important research. He, and you, will look to the recognized source of more scientifically formulated and laboratory-controlled materials—The Griffith Laboratories.

*Trade Mark Reg.
**Patent Applied For

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Stabilizing fats and oils against oxidative deterioration is a big job, too. Each molecule of antioxidant must protect, on the average, 200-500 molecules of fat. And remember, each fat molecule is subject to oxidative attack at a number of different points. Such protection must be long-lasting to protect food products and assure customer satisfaction with the protected foods.

Naturally, then, antioxidant potency is of extreme importance. BHA or butylated hydroxyanisole is a mixture of two closely related chemicals called 2-, and 3-tertiary butyl-4-hydroxyanisole. 3-BHA is far more potent than 2-BHA in antioxidant properties. Sustane 1-F has consistently been higher in 3-BHA content than the BHA of any other manufacture. Today, Sustane 1-F is higher in 3-BHA content than ever before!

It's not surprising, then, that every year more and more products are Sustane protected. Users are recognizing the leadership of Sustane in supplying more potent antioxidants, realizing **more antioxidant protection** for their **antioxidant dollar** and supplying better **Sustane protected** products to their customers.

Don't forget, either, that even the best antioxidants must be properly employed for maximum benefit. Laboratory and technical help are available so that Sustane quality is transplanted into maximum antioxidant protection in your products.

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UNIVERSAL OIL PRODUCTS COMPANY

30 EAST ALGONQUIN ROAD,
DES PLAINES, ILL., U. S. A.

FOOD INDUSTRY TOPICS

(Continued from page 30)

ous dispersion methods and other technical data. A copy may be obtained without charge.

◆ ◆ ◆

The latest Fritzsche Brothers, Inc., **prices on essential oils, aromatic chemicals, and allied products** are available in a new January 1954 Price List. Firms that buy in wholesale quantities may obtain a copy by writing Fritzsche Brothers, Inc., Port Authority Bldg., 76 Ninth Ave., New York 11, N. Y.

◆ ◆ ◆

Ninety-nine **tested candy formulations,** from kisses to candy apples, and cross-indexed by product and type of candy, are offered in a new 76-page candy recipe book, "Pocket Guide to the World's Finest Candies." Copies are available from White Stokes Company representatives or from the company's main office at 3615 S. Jasper Pl., Chicago 9, Ill.

◆ ◆ ◆

A new **vibrating feeder** that operates at near-resonant frequencies is pictured and described in data sheet No. 5302, offered by Richardson Scale Co., Van Houten Ave., Clifton, N. J. This feeder has an unusually wide operating range, being capable of turning out from 0.1 to 20 cubic feet of product per minute. The data sheet suggests uses, lists specifications, and gives a thorough explanation of operating principles. For copies write Mr. Richard Steensma at the above address.

◆ ◆ ◆

"I/TV," or **industrial television,** as distinguished from "E/TV," or entertainment television, is simmering on the back of prosperity's stove, says the January *York Report*, published by York-Gillespie Mfg. Co., 39th St. and Allegheny Valley R. R., Pittsburgh 30, Pa. The *Report* cites many uses of closed-circuit television, including the raising of the submarine *Affray*, introduction of new products by large companies, auctioning Florida fruit in a dozen cities at once, teaching surgery, checking military production, guarding prisons, verifying check signatures, controlling smoke, and watching babies. "The greatest possibilities are still to be dreamed," the *Report* concludes.

◆ ◆ ◆

The Flame Photometer, an instrument for **easy and accurate analysis of sodium and potassium,** is described in 4-page Bulletin No. 37, offered by Baird Associates, Inc., 33 University Rd., Cambridge 38, Mass. Average reproducibility of the Photometer is of the order of plus or minus 0.3% for internal standard operation. Forty or more samples an hour are frequently analyzed.

◆ ◆ ◆

A store of **new information relating to many phases of oil and fat technology** has been developed and reported by members of the Southern Regional Research Laboratory, USDA. These scientists have covered their work in 84 technical publications, published since 1941, giving information on a variety of fatty products—modified fats, mono-, di-, and triglycerides, and oils and fatty acids from cottonseed, peanuts, tung nuts, sesame seed, rice, and various minor oilseeds. An abstract bibliography of these publications (**The Physical Properties of Fatty Acids, Glycerides, and Derivatives,** compiled by W. S. Singleton, AIC-361, processed August 31, 1953) is available to research workers and processors concerned with vegetable oils or derivatives of vegetable oils. For a free copy, write the Southern Regional Research Laboratory, Southern Utilization Research Branch, Agricultural Research Service, 2100 Robert E. Lee Blvd., New Orleans, La.

◆ ◆ ◆

Seven new **Testing Procedures on the testing of fiber shipping containers** have been distributed to members of the Packaging Institute. These procedures are standardized methods of testing both corrugated and solid fiber shipping containers as recommended by the Institute's Technical Committee on Shipping Containers. While these procedures have been adapted from corresponding methods of testing by American Society for Testing Materials, in the Packaging Institute series they feature detailed introductions prepared for the benefit of the newcomer to package testing. The tests further give informa-

tion that will enable the user to interpret correctly test results. These Procedures may be purchased at $0.25 per test or $1.75 for the set of 7 tests. Write to Packaging Institute, 342 Madison Ave., New York 17, N. Y.

◆ ◆ ◆

Basic engineering data of industrial rolls, of value to textile, paper, plastics, and rubber mill engineers, are available in "Body Roll Deflections," first report of a series prepared by Rodney Hunt Machine Co., Orange, Mass. Report No. 1, listing the modulus of elasticity for 7 different materials commonly used for rolls, presents deflection equations and means of determining the moment of inertia in roll-body cross sections of hollow and solid rolls. Copies will be furnished on request.

◆ ◆ ◆

Continental Can Company forecasts in *The Florida Citrus Industry*, a recently published research study, that during the 1956-57 season, **66% of an all-time high citrus production** of 150 million boxes of fruit will be **processed**—mostly in cans. According to Continental, the six-ounce can is expected to remain the dominant container. Consumers like it because it is convenient and attractive; the retailer likes it because it can be attractively displayed and easily handled; packers like it because it can be speedily filled and frozen and it displays the brand name effectively. Copies of the study are available on request from the company's Publicity Dept., 100 E. 42nd St., New York 17, N. Y.

. . . *meeting hall and conference room*

The Refrigeration Research Foundation, 200 Midland Bldg., Colorado Springs, Colo., announces the **Refrigerated Warehousing Workshop,** April 21-22, at Chicago's Edgewater Beach Hotel. In addition to members of the Foundation and leaders of TRRF projects, Scientific Advisory Council members will also be present for their regular annual meeting. The Foundation also announces the **Annual TRRF Meeting,** to be held at Boca Raton, Fla., April 25-28. At this meeting, Dr. B. E. Proctor of Massachusetts Institute of Technology will speak on the subject of irradiation of foods, and Mr. E. Simons of San Francisco, past president of the American Society of Refrigerating Engineers, will discuss jet-air freezing of commodities.

◆ ◆ ◆

The Association of Cereal Research, in conjunction with the Association of Starch Industry, Germany, announces a **Starch Convention** to be held at Detmold, Germany (West Zone), April 26-28. Papers will be presented by leading European investigators in the areas of starch research, production and technology of starch, starch derivatives, and analysis of starch. For further information or application forms, write Dr. E. Lindemann, Assn. of Cereal Research, 9, Am Schutzenberg, Detmold, Germany.

◆ ◆ ◆

The **45th Annual Convention of the Flavoring Extract Manufacturers' Association of the United States** is scheduled for the Hotel Biltmore in New York City, May 16 through 19. Mr. Wm. H. Hottinger, Jr., Bowey's Inc., Association president, and all members of the FEMA Board of Governors are assisting the Convention Committee, headed by Mr. Robert Krone, Fritzsche Brothers, Inc. The business program will feature discussion by outstanding management, sales, and production authorities.

◆ ◆ ◆

The **45th Annual Meeting of the American Oil Chemists' Society,** to be held in San Antonio, Texas, April 12-14, will be in charge of Judson S. Swearingen, Southwest Research Institute. Program chairman will be W. D. Harris, Texas A&M College. The AOCS also sends word that the subject of the August "short course," scheduled for Lehigh University, will be "Inedible Fats and Fatty Acids" (except soap and drying oils). Chairman will be Daniel Swern of the Eastern Regional Research Laboratory, USDA, Philadelphia.

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INSTITUTE OF FOOD TECHNOLOGISTS
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Chicago 3, Illinois

FOOD INDUSTRY TOPICS

. . . *building up and branching out*

An **Ammonia Division,** to handle the manufacture, distribution, and sale of this chemical vital to agriculture and industry, was initiated effective January 1, 1954, by Shell Chemical Corp., New York City. Headquarters of the new division will be San Francisco. The company's ammonia plant was opened at Ventura, Calif., on December 11, 1953. In charge of the new operation is Mr. G. R. Monkhouse, Shell vice-president. Mr. L. M. Roberts, general manager of manufacturing, is the new division's operations manager in charge of manufacturing, distribution, and marketing engineering. Mr. V. C. Irvine has been designated division sales manager. In addition to their new duties, Mr. Monkhouse and Mr. Irvine will continue as manager and sales manager, respectively, of Shell Chemical's Western Division, which handles sales of all company products.

◆ ◆ ◆

Distributorship for the Westfalia clarifier, a versatile laboratory centrifuge that can be used for clarification, mixing and emulsifying, solvent extraction, separation, and concentration of solids, has been announced by Cherry-Burrell Corp., 427 W. Randolph St., Chicago 6, Ill. Small and light, the instrument is reportedly ideal for use in research, pilot plant operations, or quality control.

. . . *the human element*

Magnus, Mabee & Reynard, Inc., essential oil firm, announces appointment of **Mr. Bernard Jeffs,** formerly vice president in charge of sales and advertising for Reed & Carnick, as director of advertising. This appointment, stated Mr. Percy C. Magnus, MM&R president, is synchronized with a broad program of research and development to facilitate further expansion in world markets.

◆ ◆ ◆

Promotion of Assistant Editor **Karl Robe** to associate editor was announced by Mr. Howard Milleville, editor of *Food Processing,* Chicago. Mr. Robe holds membership in national science, agricultural, and journalism fraternities, as well as an active membership in the IFT.

◆ ◆ ◆

Mr. Bruce Lister, an IFT member, has been promoted to assistant laboratory director, product and process development, at General Foods Central Laboratories, Hoboken, N. J., it was announced by Mr. Harvey K. Murer, laboratories manager.

◆ ◆ ◆

Election of **Mr. Wm. Geisler** as executive vice president of National Phoenix Industries, Inc., New York City, has been announced by Mr. Walter S. Mack, president. Mr. Geisler, an IFT member, is vice president of Nedick's, Inc., and of Cantrell & Cochrane Corp., and is a director of both firms as well as of National Phoenix Industries. During World War II, he served as technical adviser to OQMG in food packing and production problems.

◆ ◆ ◆

Mr. Thomas J. Herman has joined the sales staff of Givaudan-Delawanna, Inc., and its associate companies, Givaudan Flavors, Inc., and Sindar Corp., according to announcement by Mr. R. E. Horsey, vice president in charge of sales of Givaudan-Delawanna. The new sales representative will service accounts in the territory covered by the Cincinnati branch office.

◆ ◆ ◆

Appointment of **Mr. R. B. Thompson** as assistant general manager of manufacture for the American Can Company has been announced by Mr. G. W. Reese, general manager of manufacturing. Mr. Thompson, previously manager of manufacture for the company's Atlantic Division, has been with the container firm for 31 years. He will continue to make his headquarters in New York. The company also announced that **Mr. A. de Genaro,** formerly assistant manager of manufacture for the Atlantic Division, will succeed Mr. Thompson as division manager.

Choose from fmc's FAMOUS FAMILY OF M & S FILLERS

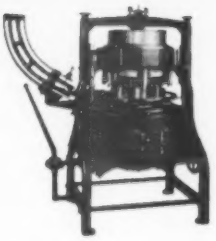
The Standard for Progressive Canners because they are UNSURPASSED for ACCURACY

FMC M&S Plunger Fillers have set new standards for high accuracy filling. On the basis of thousands of accuracy of fill tests, by weight testing methods, M&S Fillers are *unsurpassed for accuracy!*

And M&S Fillers lead the field for *versatility,* too: no other fillers can handle as wide a range of liquid, semi-liquid and heavy semi-solid products.

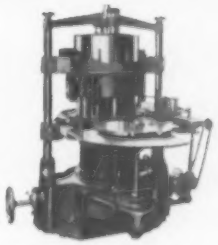
These fillers are engineered for heavy duty and long life. Operating parts are fully enclosed. Drive gears run in oil. All inspection, lubrication, and adjustment points are readily accessible. Non-corrosive metals are used on all contact surfaces. Special "No-Can, No-Fill" feature prevents product waste.

By all comparisons, these are the finest fillers made — designed and built to perform better at lower cost.



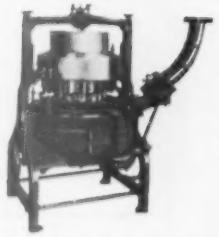
FMC M & S 6 Pocket Fillers

Handles virtually any liquid, semi-liquid or semi-solid product. Can be furnished for can sizes from 202 x 102 to 610 x 910. Speed: 150 CPM in small containers.



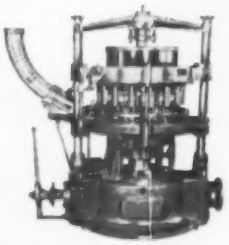
FMC M & S 9 Pocket Fillers

For filling pulps, pastes and liquids in containers from 202 x 203 to 404 x 611 size. Speed: up to 150 CPM on light liquids; 250 CPM on heavier products.



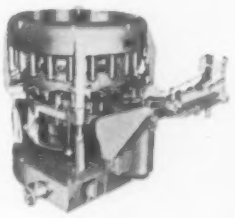
FMC M & S 10 Pocket Fillers

Handles a host of products of varying consistencies from condensed milk to heavy dog food for containers ranging in size from 202 x 102 to 404 x 604. Speeds up to 300 CPM.



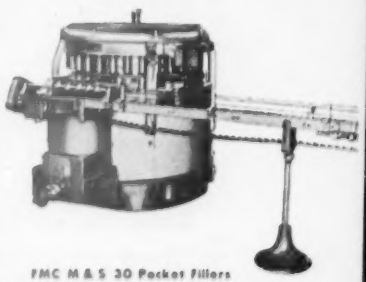
FMC M & S 15 Pocket Fillers

For container sizes up to 211 x 400. Handles citrus concentrates, baby foods and other semi-liquid free flowing products at up to 450 CPM.



FMC M & S 20 Pocket Fillers

Delivers up to 500 CPM using jars or cans. Typical products filled are lubricating oil, apple sauce, and chocolate syrup. Handles containers within a range of 202 to 401 diameter.



FMC M & S 30 Pocket Fillers

Designed for baby foods and similar products. Handles 5 to 8 ounce jars or cans at speeds of 600 CPM.

There's an M & S Filler for virtually every product, can size, and can line speed

Write for full information, or call your nearest FMC representative.

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FUTURE DATES FOR FOOD TECHNOLOGISTS

1954	
March 10-12	International Meeting of the American Society of Mechanical Engineers, Mexico City, Mexico
April 22-23	National Meeting of the American Dry Milk Institute, Edgewater Beach Hotel, Chicago, Ill.
April 25-28	Annual Meeting of The Refrigeration Research Foundation, Boca Raton, Florida
April 27-28	Annual Meeting of the National Cheese Institute, Chicago, Ill.
May 2-5	Annual Meeting of the National Fisheries Institute, Cleveland, Ohio
May 16-19	Flavoring Extract Manufacturers' Association, Biltmore Hotel, New York, N. Y.
May 19	Fruit and Syrup Manufacturers' Association, New York, N. Y.
May 26-28	*Fourth Annual Convention, Australian Regional Sections, Institute of Food Technologists, Leura, New South Wales, Australia.*
June 23-25	Seventh Annual Meeting of the Research and Development Associates, Food and Container Institute, Los Angeles, California
June 27 to July 1, 1954	**Fourteenth Annual Meeting of the Institute of Food Technologists, Los Angeles, California**

(Information concerning future dates of national and international meetings, conventions and exhibits of interest to food technologists should be sent to the editorial office.)

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AVAILABLE: Food Technologist, B.S. in Food Technology. Experience in frozen foods, canning, experimental work, and quality control. Desire responsible position in research and development in the New England area. Would consider production and quality control. REPLY BOX 316, Institute of Food Technologists, 176 W. Adams Street, Chicago 3, Ill.

CANDY TECHNOLOGIST WANTED—Should possess background in analytical procedures and techniques. The Food Technologist hired for this position will be requested to act as liaison between the laboratory and the buying office of a general line candy manufacturer firm. REPLY BOX 319, Institute of Food Technologists, 176 W. Adams Street, Chicago 3, Ill.

AVAILABLE: Food Technologist with experience in diversified line of products and types of operations (canning, freezing) and academic background in business. Desires to invest and participate in small to medium size food manufacturing organization. REPLY BOX 317, Institute of Food Technologists, 176 W. Adams Street, Chicago 3, Ill.

AVAILABLE: Food Technologist, B.S. 41 yrs. 12 yrs. experienced canned and frozen vegetables and citrus. Production, quality control, research and technical service. Prefer West Coast. REPLY BOX 318, Institute of Food Technologists, 176 W. Adams Street, Chicago 3, Ill.

AVAILABLE: Food Technologist, Bacteriologist, Chemist, Sales Minded. M.S. plus. Married. Age 48. 15 yrs. diversified food experience. Quality Control, Flavor Expert, Selecting Ingredients, Processing Development, Purchasing, Writing, Toppings, Beverage Bases, Dry Mixes, Sanitation, Salad Dressing, Creative, Down to Earth, New Thinking. REPLY BOX 315, Institute of Food Technologists, 176 W. Adams Street, Chicago 3, Ill.

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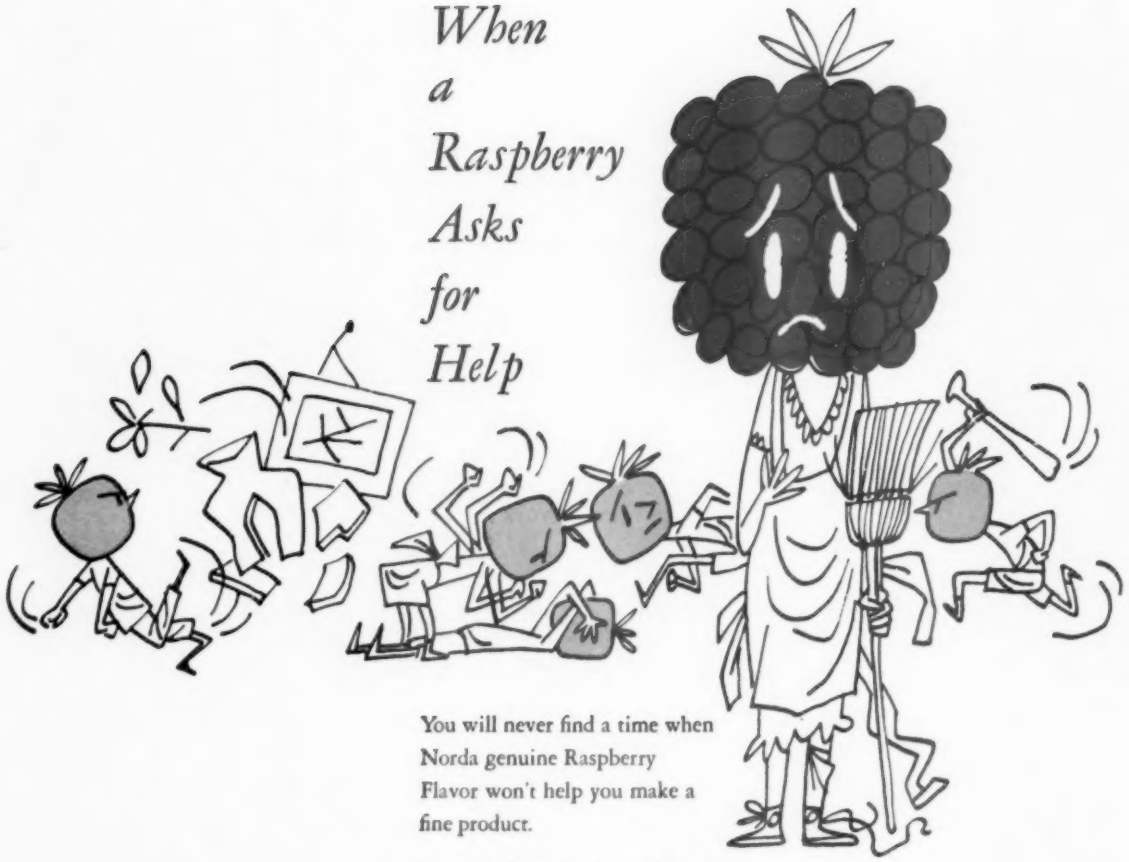

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